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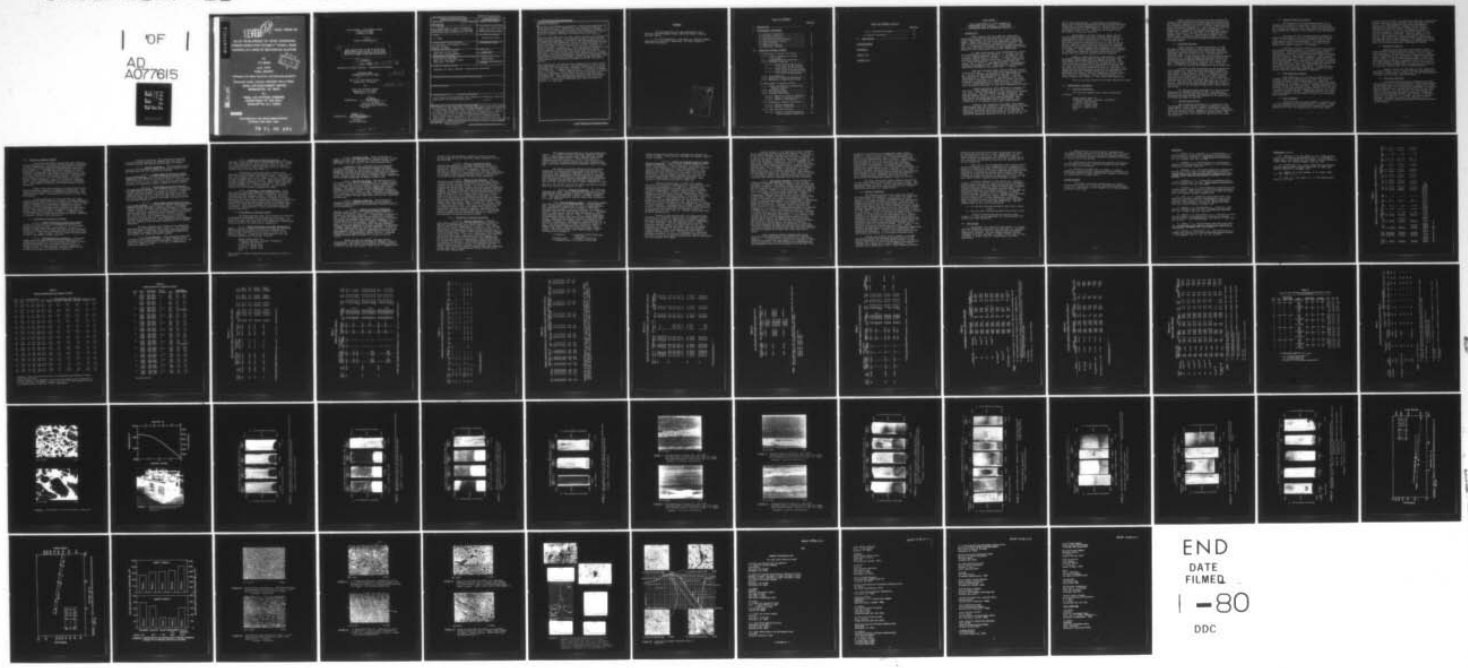

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ALLOY DEVELOPMENT OF OXIDE DISPERSION STRENGTHENED HIGH VOLUME γ' NICKEL-BASE SUPERALLOYS MADE BY MECHANICAL ALLOYING

by

R.C. BENN

July 1979

FINAL REPORT

"APPROVED FOR PUBLIC RELEASE - DISTRIBUTION UNLIMITED"

Prepared Under Contract N62269-78-C-0200

NAVAL AIR DEVELOPMENT CENTER
WARMINSTER, PA. 18974

For

NAVAL AIR SYSTEMS COMMAND
DEPARTMENT OF THE NAVY
WASHINGTON, D.C. 20361

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The object of this work was to develop ODS alloys which would derive a significant high temperature strength increment from the retention of high volume fractions (>50%) of (γ') at 2000°F (1093°C). Alloy structure and property investigations were conducted to determine the effects of W, Mo, Ta and Nb combinations substituted on an atomic weight basis into each of three Ni-Cr-Al ternary systems. Specifically, Cr:Al levels of 15:16, 12.5:17.5 and 10:17.5 at.% were chosen. Based on promising directional recrystallized (Cont'd)

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structures and mechanical properties, three alloys were selected from the series of 12 compositions, for further development including the effects of B/Zr alloy modifications, extrusion, hot rolling and heat treatment studies on the directional recrystallization response, structure and mechanical properties.

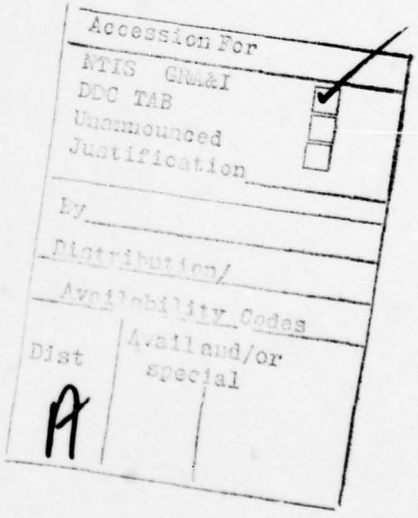
The work confirmed that the high volume fraction of γ' (gamma prime) characteristic of these alloys, is an effective means of improving the intermediate temperature strength of ODS superalloys. A Ni-Cr-Al-W-Mo-Ta-Nb ODS composition, Alloy 49, was identified from the three selected alloys, having 100-hour rupture strengths of 90 ksi (620.5 MPa) at 1400°F (760°C) and 19.5 ksi (134 MPa) at 2000°F (1093°C), respectively. The intermediate strength of this alloy surpassed that of MA 6000E (80 ksi/552 MPa) and was equivalent to high strength cast alloys such as IN-100 (91 ksi/627 MPa). The alloy thus achieved the combination of excellent intermediate and high temperature strength in a composition range known from previous work (NAVAIR contract N62269-76-C-0483) to have superior hot corrosion resistance to IN-100, IN-713C or B-1900.

The directional recrystallization response of the three alloys was improved by hot rolling. Maximum strength in Alloy 49 was developed with a simple heat treatment and structural analyses confirmed the γ/γ' phase field location. A correlation between the γ' content, γ' solvus temperature and directional recrystallization temperature of the three alloys was observed which suggested that minor compositional changes would be beneficial to properties. Detailed characterization of the final high volume % γ' alloy can be addressed once these optimization studies are completed.

FOREWORD

The work reported here was performed by Inco Research and Development Center (Sterling Forest, Suffern, New York 10901).

Mr. Irving Machlin of the Naval Air Systems Command (Department of the Navy, Washington, DC 20361) acted as technical consultant.



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FINAL REPORT

ALLOY DEVELOPMENT OF OXIDE DISPERSION STRENGTHENED HIGH VOLUME % γ' NICKEL BASE SUPERALLOYS MADE BY MECHANICAL ALLOYING

I. INTRODUCTION

Today, several distinct types of alloys with directional structures offer potential for increased operating temperatures (100-350°F) for gas turbine vanes and blades. Among these are the oxide dispersion strengthened (ODS) alloys(1-3) produced by mechanical alloying(4). The ODS + γ' nickel-base superalloys which have been produced to date offer substantial increases in high temperature strength capability over conventional nickel-base superalloys.

In these previous ODS alloy investigations, alloys with conventional volume fractions of γ' (30-50%), and low γ' solvus temperatures were studied. At intermediate temperatures (1400°F[760°C]), the ODS + γ' alloys enjoy a significant strength increment over their γ'-free counterparts; e.g., TD-Ni and TD-Ni-Cr. As the use temperatures are raised, the γ' solvus is approached and the γ' strengthening increment decreases to zero. This occurs at approximately 1600-1800°F (870-980°C) for present ODS + γ' alloys.

Work was initiated under Contract N00019-75-C-0313 to develop an ODS + γ' alloy with a very high volume fraction of γ' at the intended use temperature of 2000°F(1095°C). The intention of that program was to combine γ' and ODS strengthening at 2000°F(1095°C). The results of this previous work(5) show that it is possible to produce an oxide dispersion strengthened high volume fraction γ' (90%) Ni-Cr-Al alloy, designated alloy 2, with the coarse elongated grain structures necessary for good high temperature strength.

Through quaternary alloy additions of tungsten to the alloy 2 base composition (Ni-10 wt.% Cr-9 wt.% Al-1.1 wt.% Y_2O_3), further improvements in high temperature strength were achieved. Despite a small increase in density, the highest tungsten alloy 8 ($\sim$7 wt.% W) showed superior 1000-hour density corrected specific rupture strength, when compared to the DS MAR-M200 (above 1530°F[830°C]) and a current DS γ/γ'-α eutectic alloy (above 1630°F[890°C]).

Based on the feasibility and modest improvements demonstrated under that contract, a new program was initiated under NAVAIR Contract N62269-76-C-0483 to investigate the effects of quaternary and higher order alloy additions on the properties of the Ni-Cr-Al ODS alloy. Specifically, the effects of quaternary additions of W, Ta, Nb, Mo, Co, Hf

and Ti were investigated. In particular, the quaternary alloys with W and Mo respectively exhibited significant high temperature strength improvements(6). Raising the Cr level in the ternary Ni-Cr-Al base alloy to 15 wt.% reduced the sulfidation attack by 50%. Oxidation resistance of the experimental alloys was excellent.

Subsequently, the data from the simple ternary and quaternary alloys were used to design more complex alloys based on atomic substitution in the more corrosion resistant Ni-15 wt.% Cr-9 wt.% Al-1.1 wt.% Y_2O_3 alloy. The alloys contained combinations of W, Co, Mo, Ta, Nb, Ti and Hf. However, these alloys did not yield the required directionally recrystallized grain structure on gradient annealing. The poor response was attributed to the presence of β phase(6). Minor compositional modifications, designed to overcome this problem, evolved a promising series of alloys with the required recrystallization response.

Based on these progressive improvements, a follow-on research program has been initiated to continue alloy development under NAVAIR Contract N62269-78-C-0200. The first objective of this program was to complete the evaluation of the modified series of more complex alloys so that the three most promising alloy systems can be selected in the light of alloy data generated to date in this work. The second and main objective was to develop the thermomechanical processing and heat treatment conditions to optimize the properties and enable a critical comparison of these alloys. This work will, ultimately, lead to the development of a single complex high volume % γ' ODS nickel base superalloy.

This is the final report for Contract N62269-78-C-0200.

II. EXPERIMENTAL PROCEDURES

2.1 Attritor Processing

The following powders were used for mechanical alloying:

Nickel powder Type 123
Elemental chromium, tungsten, molybdenum, tantalum and niobium
Ni-47.5 Al master alloy
Ni-28 Zr master alloy
Ni-16.9 B master alloy
Y_2O_3

Powder batches of these alloys were mechanically alloyed in the attrition mills under controlled conditions. The resultant mechanically alloyed powder was characterized using chemical analysis for O, N, C and Fe, screen analysis and metallographic examination. The criteria for accepting powder as well processed are outlined in Reference 4. Essentially, a powder is considered processed when metallographic examination indicates that it is completely homogeneous (e.g., Figure 1). Experience has taught that a coarse powder size distribution, normal oxygen (~0.5-0.8 wt.%) and iron (0.5-0.8 wt.%) levels are indicative of well processed powder.

2.2 Extrusion Practice

After screening to remove the coarse +12 mesh particles, powder batches of each composition were cone blended for 2 hours and packed into mild steel extrusion cans which were then sealed in air. Previous work(6) was conducted using 3.0 in (7.6 cm) ID/3.5 in (8.9 cm) OD extrusion cans. A preliminary study in the present contract, using Alloy 37, showed no apparent difference between the recrystallization response obtained with 0.5 in (1.3 cm) wall (Batch V121) and 0.25 in (0.64 cm) wall (Batch V123) cans. Consequently the more standard 3.25 in (8.25 cm) ID/3.5 in (8.9 cm) OD cans were adopted in the present work.

A minimum of four extrusion cans was prepared for each composition. Extrusions were made after preheating the billets in the range 1-2 hours at temperatures in the range 1850°F (1010°C)-2200°F (1205°C). Conical extrusion dies having an included angle of 90° were used yielding round bar (0.8 in/20.4 mm ϕ) at an 18:1 ratio, while rectangular dies were used to extrude bar (1.2 in x 0.8 in/30.2 mm x 20.4 mm) at a 10:1 ratio for subsequent hot rolling trials. Lubrication was provided by a glass pad on the die face with oil in the extrusion chamber and a glass wrap on the billet.

All extrusions were performed on a 750 ton Loewy Hydropress at throttle settings of 35%. Ram speed and pressure were continuously recorded during each extrusion. Occasional recorder malfunction resulted in no record for a few extrusions made in this work.

2.3 Hot Rolling Practice

The rectangular extruded bar of selected alloys was preheated 1 hour and hot rolled at temperatures in the range 2050°F (1120°C)-2250°F (1230°C) with height reduction levels of 20, 40 and 60%. Reheats of 10-15 min at temperature were given between the 20% reduction rolling passes. The hot rolled bars were then pickled to remove the mild steel extrusion can.

2.4 Gradient Annealing Studies

The objective of the extrusion studies was to determine the conditions required to yield a coarse elongated grain structure, in each alloy, upon heat treatment. The necessity of obtaining this structure to achieve maximum high temperature strength in ODS alloys is well documented (7,8).

The nature of the recrystallization response is generally described in terms of transverse grain diameter (fine .1-1 μm, medium 1-50 μm and coarse 50-250 μm) and grain aspect ratio (length/diameter). For optimum high temperature strength, a coarse grain size with a grain aspect ratio (GAR) of about 10 is required.

Stationary gradient anneals were used to determine the recrystallization behavior as a function of annealing temperature. Four inch long extruded bar samples were individually annealed in a gradient furnace having a thermal profile similar to that shown in Figure 2. Total annealing time was 1/2 hour. After heat treatment, these bars were surface ground parallel to the extrusion direction to reveal the recrystallized grain structure as a function of position along the gradient. This is a simple method for determining the temperature range over which recrystallization to a coarse grain structure will take place. It also pinpoints the critical heat treatment temperature at which the best coarse elongated grain structure is achieved.

2.5 Zone Annealing Studies

Zone annealing (Z.A.) can be an effective way of increasing the grain aspect ratio and hence the high temperature strength of ODS superalloys(1,9). Generally, extruded bar which shows a coarse grain structure (elongated or equiaxed) on isothermal or static gradient annealing will respond favorably to zone annealing. Selected bars were zone annealed in the same gradient furnace (Figure 2) at 2.8 iph (7.1 cmph) and maximum zone temperatures ranging from 2225°F (1220°C) to 2420°F (1330°C). Specimens were cut from these zone annealed bars and sectioned to reveal the grain aspect ratio achieved.

2.6 Heat Treatment

Selected alloys were heat treated to determine the γ' solvus temperature and volume percent γ' present. These data were derived from microstructural studies of material soaked for 3/4 hour at temperatures in the range 2050°F

(1120°C)-2390°F (1310°C) and then water quenched. Appropriate solution and aging heat treatments were then applied to the alloys. The metallographic studies were subsequently complemented by Differential Thermal Analyses (DTA). Specimens for DTA were melted in Al_2O_3 or Pt crucibles into which a Pt vs. Pt/13% Rh thermocouple extended. A similar arrangement using high purity Ni powder in a Pt crucible served as a reference. Heating ranges of 10 or 20°C/min were used with a high purity argon flush of 50 ml/min. The differential output between the thermocouples was expanded using a DCmV amplifier. The sample was melted and then remelted, since transformation characteristics were found to be more pronounced on reheating the sample.

2.7 Mechanical Testing

Specimens for mechanical testing were ground from round heat treated bars with their tensile axis orientated parallel to the extrusion direction. In all cases this corresponded to the direction of structural elongation. Stress rupture tests were performed at 1400°F (760°C) and 2000°F (1093°C). These tests were performed in accordance with ASTM specifications on specimens with 0.125 in (3.18 mm) gauge diameter, gauge length of 1 in (25.4 mm) and 0.25 in (6.35 mm)-20NC threaded ends. Elongation and reduction of area were measured from the fractured specimens.

Tensile testing was performed at room temperature and 1400°F (760°C) in air. Again, the test specimens were identical in dimension, orientation and method of manufacture to those used for stress rupture testing.

2.8 Structural Analyses

A detailed microstructural examination was made using optical and electron microscopy. Phase identification was achieved using x-ray diffraction on extracted residues. Anodic dissolution in an electrolyte consisting of 1 percent ammonium sulfate and 1 percent tartaric acid in water was used to extract γ' and any β-NiAl. This electrolyte will also extract carbides, nitrides and borides. Berzelius reagent ($CuCl \cdot 2H_2O/KCl$/tartaric acid solution in HCl/H_2O), which will not extract γ' or β-NiAl, was used to detect carbides, nitrides, oxides and any σ-phase or α'(Cr). Electron microprobe analysis was used to identify optically visible phases.

III. TECHNICAL PROGRESS SUMMARY

This alloy development program has been conducted in two parts based on the results of the previous contract(6). Firstly, compositional modifications were made to eliminate the formation of any undesirable β-phase while retaining a high volume % γ′. The modified alloys (Series III) were designed to investigate the effects on properties of W, Mo, Ta and Nb combinations substituted on an atomic weight basis into each of three Ni-Cr-Al ternary systems. Specifically, Cr:Al levels of 15:16, 12.5:17.5 and 10:17.5 at.% were chosen. These represent a range in sulfidation resistance due to Cr(6), while maintaining the Al level at a maximum for γ′ strengthening consistent with phase diagram considerations(10).

These alloys were screened in terms of directional recrystallization response and mechanical properties. Good stress rupture properties were obtained in preliminary step-load tests such that it was possible to select an alloy from each system.

In the second part of this contract, the three selected alloys were evaluated in detail, including mechanical testing and the effects of B and Zr additions on the recrystallization response and intermediate temperature strength. In addition, extrusion and hot rolling studies were made to characterize the thermomechanical properties of these three alloys. The effects of heat treatment on properties and microstructure were evaluated in detail. A general review of the alloy data was made and optimization studies commenced to isolate a single alloy composition.

3.1 Evaluation of Series III Alloys

3.1.1. Powder Processing of Series III Alloys. A list of the Series III alloys prepared by mechanical alloying is given in Table I. Two 18.7 lb (8.5 kg) powder batches of each composition were produced. Prior to a change in composition, one wash heat was made and discarded. The results of the powder characterization studies are given in Table II. Figure 1 shows a typical etched microstructure of one of the powder batches (Alloy 42).

3.1.2. Thermomechanical Processing. After each powder batch had been qualified as well processed using established criteria(4), the two batches were mixed by cone blending and packed into mild steel extrusion cans. A minimum of four cans was prepared for each composition listed in Table I. The extrusion conditions used for consolidation of powder are detailed in Table III.

Following extrusion, the alloys were tested for recrystallization response by gradient and zone annealing using the furnace and thermal profile shown in Figure 2.

3.1.3. Gradient Annealing. The recrystallization response was evaluated in terms of the alloys derived from the three Ni-Cr-Al ODS ternaries as follows:

3.1.3.1. Alloys Based on Ni-15 at.% Cr-16 at.% Al ODS Ternary. The most promising recrystallization response in this system of alloys was shown by Alloy 38 which contains 2 at.% (6.5 wt.%) W, 1 at.% (1.7 wt.%) Mo and 0.5 at.% (1.6 wt.%) Ta. Figure 3 shows that a high GAR was achieved in recrystallized bar extruded at 2150°F (1177°C).

3.1.3.2. Alloys Based on Ni-12.5 at.% Cr-17.5 at.% Al ODS Ternary. Bar V138D of Alloy 42, containing the same levels of W, Mo and Ta as Alloy 38 gave an excellent recrystallization response, as shown in Figure 4. A more complex alloy (Alloy 44), containing the same levels of W and Mo but with 1 at.% (3.2 wt.%) Ta and 1 at.% (1.6 wt.%) Nb, also yielded the required directional recrystallization in bar V144D (Figure 5).

3.1.3.3. Alloys Based on Ni-10 at.% Cr-17.5 at.% Al ODS Ternary. In view of the lack of recrystallization response in bars extruded at 1850°F (1010°C) in the above alloys, material from this system was extruded at 1950°F (1065°C) and above. Alloy 47, as shown in Figure 6, exhibited the most promising recrystallization response in this system. This quinary alloy, containing 2 at.% (6.6 wt.%) W and 2 at.% (3.4 wt.%) Mo, was a combination of the promising quaternary alloys with W and Mo, respectively, that were evolved in previous work(6). Some recrystallization response was also shown by Alloy 48 which contained Mo, W, Ta and Nb additions.

The best response with alloys in this system was obtained on bar extruded at 2150°F (1177°C). This observation was common to the other two ternary systems and there was evidence to suggest that higher extrusion temperature may enhance the recrystallization response.

3.1.4 Zone Annealing. Zone annealing conditions of 2360°F (1295°C) and 2.8 iph (7.1 cmph) produced good directionally recrystallized structures in Alloys 38, 42, 44 and 47, as shown in Figure 7, 8, 9 and 10, respectively, with GAR's generally >15.

3.1.5. Mechanical Property Evaluation. Preliminary stress rupture data on as-extruded and heat treated bars were generated for the promising Alloys 38, 42, 44 and 47. Step-load stress rupture results, given in Table IV, showed that Alloys 44 and 47 had lives of 15.8 h and 9.2 h, respectively, at 20 ksi and 2000°F.

Conventional fixed load stress rupture tests were then run on the best three alloys (i.e., Alloys 38, 44 and 47) at 1400°F (760°C) and 2000°F (1093°C). The bars were zone annealed and given a simple 1/2 h/Z.A. temperature/AC heat treatment prior to testing. These results are given in Table V which indicates that the 1400°F (760°C)/100-hour rupture strength of all three alloys was at least equal to that of MA 6000E, i.e., 80 ksi/552 MPa. In particular, Alloy 44 (85 ksi/586 MPa) surpassed MA 6000E.

3.1.6. Selection of Best Three Alloys. Since each ternary system was based on a different Cr:Al ratio, the three systems covered a range of potential corrosion resistance(6) and strength levels. Alloys 38, 44 and 47 were selected for further evaluation, primarily for their promising recrystallization response and mechanical properties. Coincidentally, these three alloys were also derived from each of the three ternary systems so that a desirable range of potential corrosion resistance and strength levels was maintained. This presented a wider choice for subsequent alloy optimization.

3.2 Development of Selected Alloys

The remaining work under this contract was directed at developing the properties of Alloys 38, 44 and 47. In particular, the effects of minor B/Zr additions (which are known to enhance the properties of MA 6000E) were investigated using Alloy 44 as the main control composition.

3.2.1. Powder Processing of Alloys Containing B and Zr. The B/Zr modified versions of Alloys 38, 44 and 47 were identified as Alloys 50, 49 and 51, respectively. These alloys contained 0.01 wt.% B and 0.15 wt.% Zr, as shown in Table VI. These alloys were prepared using the following powders for mechanical alloying:

Nickel powder Type 123
Elemental chromium, tungsten, molybdenum, tantalum and niobium
Ni-47.5 Al master alloy
Ni-28 Zr master alloy
Ni-16.9 B master alloy
Y_2O_3

The results of powder characterization studies are given in Table VII.

3.2.2. Extrusion Study. After screening to remove the coarse +12 mesh particles, powder batches of each composition were cone blended for 2 hours and packed into mild steel 3.25 inch I.D./3.5 inch O.D. extrusion cans.

A minimum of four extrusion cans was prepared for each composition. Following preliminary successful extrusions of Alloy 49 (Batch V159), a matrix of extrusion parameters centered on the successful conditions was examined to enlarge the "recrystallization response window" of each alloy. Specifically, the effects of preheat time, temperature and ratio were determined as shown in Table VIII.

3.2.3. Hot Rolling Study. The objective of the hot rolling trials was to investigate the hot workability of the alloys, since it is anticipated that an ultimate production route for the alloys as turbine blades would include forging. The effects of hot rolling temperature versus percentage thickness reduction were investigated using the 1.19 in. x 0.81 in. (30 mm x 21 mm) rectangular extruded bar (10:1 extrusion ratio) of each alloy. This matrix arrangement is given in Table IX which identifies the hot roll conditions for each bar.

3.2.4. Gradient Annealing. The directional recrystallization response of the alloys was studied in relation to the effects of B/Zr additions, extrusion and hot rolling parameters.

3.2.4.1. Effect of B and Zr Additions. Figures 11 and 12 show the recrystallization response of the B/Zr modification (i.e., Alloy 49) of Alloy 44 as a function of extrusion variables. A comparison of Figures 11 and 5 (Alloy 44) shows that the particular B/Zr addition levels did not significantly effect the extrusion conditions for optimum directional recrystallization response, i.e., 2150°F (1175°C/1-1/2 h preheat. However, the temperature zone in which the desired directional recrystallization response occurred appeared to be both extended and displayed to higher temperatures with the B/Zr additions. Figure 11 also indicates that the solidus temperature of Alloy 49 was approximately 2410°F (1320°C), while that of Alloy 44 was determined to be approximately 2450°F (1345°C). Detailed thermal analysis data on Alloy 49 is given in Section 3.2.7.

Alloys 50 and 51 displayed the same general characteristics as Alloy 49, as shown in Figures 13 and 14, respectively. The extent of directional recrystallization at 2150°F (1175°C) was related, inter alia, to compositional

effects; note the excellent response of Alloy 51 which, unlike Alloys 49 and 50, does not contain elements such as Ta and/or Nb.

3.2.4.2. Effect of Extrusion Ratio. Reducing the extrusion ratio and, thereby the degree of primary hot deformation, generally decreased the level of directional recrystallization response. This is readily apparent in Figures 12-14. Secondary hot working, such as hot rolling of the 10:1 ratio sheet bar, was then investigated to introduce sufficient deformation energy to improve the recrystallization response and GAR on zone annealing.

3.2.4.3. Effect of Hot Rolling. The selection of hot rolling temperatures in the range 2050°F (1120°C)-2250°F (1230°C) was based on the recrystallization response of the extruded alloys. The effects of the hot rolling conditions given in Table IX on the recrystallization response of the bars were observed through gradient annealing studies. The most significant directional recrystallization response was obtained in bars rolled at 2050°F (1120°C), as shown in Figure 15. This figure shows that, for a given alloy such as Alloy 49, a progressive improvement in the extent of directional recrystallization and GAR was obtained with increasing secondary hot rolled thickness reduction: Compare bar V163B (0%) in Figure 12 with bars V163B-1 (20%), V163B-2 (40%) and V163B-3 (60%) in Figure 15. Alloys 50 and 51 showed the same trends. Also note, as observed with the as-extruded material, how alloy composition effected the directional recrystallization response. Specifically, in Figure 15, note how increasing additions of γ' and carbide-forming elements such as Ta and Nb reduced the extent of directionally recrystallized grain growth.

3.2.5. Mechanical Property Evaluation

3.2.5.1. Rupture Properties. Constant load stress rupture tests on extruded and heat treated bar were run to characterize Alloy 49 at 1400°F (760°C) and 2000°F (1093°C) for comparison with Alloy 44. The results are given in Table X and plotted in Figures 16 and 17. The rupture data is summarized in Table XI and indicates that Alloy 49 exhibited a 1400°F (760°C)/100-hour rupture strength of 90 ksi (620.5 MPa). This is an improvement over MA 6000E (80 ksi/552 MPa) and is equivalent to high strength cast alloys such as IN-100 (91 ksi/627 MPa), and compares well with fully heat treated directionally solidified MAR-M200 + Hf (105 ksi/724 MPa)(11). Preliminary rupture tests on the extruded bar of Alloys 50 and 51 (B/Zr modified versions of Alloys 38 and 47), given in Table X, indicated that the 1400°F (760°C)/100-hour rupture strengths were also increased by B/Zr additions to approximately 84 ksi/579 MPa and 86 ksi/593 MPa, respectively.

Some strength improvements in the high temperature region, i.e., 2000°F (1093°C) and above, where ODS rather than γ' strengthening predominates, were obtained through improved grain shape control. Specifically, Alloy 51 exhibited the best rupture life (i.e., 53.5 h/20 ksi) at 2000°F (1093°C) primarily because this alloy exhibited the best directional recrystallization response (refer to Figure 15).

The density-corrected 100-hour rupture strength properties of various materials systems were determined (Table XII) and plotted for comparison in Figure 18. This plot shows that experimental ODS Alloy 49 has an excellent combination of intermediate and high temperature strength with the potential for further improvement with appropriate heat treatment and secondary hot working, respectively. Note that on a $\frac{\sigma}{\rho}$ basis, the 1400°F (760°C) strength of Alloy 49 (311×10^3 in/775×10^3 cm) is almost the same as DS MAR-M200 + Hf (336×10^3 in/838×10^3 cm).

3.2.5.2. Tensile Properties. The room temperature and 1400°F (760°C) tensile test results on all the experimental alloys indicated superior strength advantages over IN-100 and DS MAR-M200 + Hf(12), as shown in Table XIII. The room temperature modulus values were indicative of a [110] texture.

3.2.6 Heat Treatment Studies. Preliminary studies were made on Alloy 44 to determine the γ' solvus temperature. Samples were soaked for 3/4 hour at temperatures in the range 2050°F (1120°C)-2375°F (1302°C), then water quenched and examined by optical microscopy. As shown in Figures 19 and 20, the high volume fraction of γ', characteristic of these alloys, did not begin to resolution until approximately 2300°F (1260°C). This γ' solvus temperature is considerably higher than that of many established superalloys. These data were used to design a program of heat treatment schedules to optimize the γ' precipitation in relation to properties. Extruded bar stock of Alloys 38, 44 and 47 was zone annealed at 2390°F (1310°C)/2.8 iph (7.1 cmph), while Alloy 49 bar was zone annealed at 2370°F (1300°C) and 2340°F (1280°C)/2.8 iph (7.1 cmph), respectively. The bars were then given combinations of the following heat treatments:

A (Solution : 2 h/2360°F (1295°C)/Fast Air Cool (FAC)
B(Primary Age) : 4 h/2060°F (1130°C)/AC
C (Secondary Age): 24 h/1660°F (905°C)/AC

Stress rupture test pieces were subsequently machined and tested at 1400°F (760°C)/85 ksi (586 MPa) and 2000°F (1093°C/ 20 ksi (138 MPa.

3.2.6.1. Effect of Microstructure on Stress Rupture Properties. Better stress rupture properties were generally obtained in these alloys with the simple 1/2 h/Z.A. temperature/A.C. heat treatment, rather than combinations of the treatments A, B and C defined above. For example, Alloy 49 exhibited a 1400°F (760°C)/85 ksi (586 MPa) rupture life of 182 h with the simple 1/2 h/2370°F (1300°C)/A.C. heat treatment, whereas 124 h life was the best achieved through combination heat treatments of 2 h/2360°F (1295°C)/FAC + 24 h/1660°F (905°C)/A.C. Full details of the results are given in Table XIV.

Optical metallography of broken test pieces indicated that the grain morphologies of the respective test pieces were comparable (i.e., GAR >10)). However, the microstructural features revealed by electron microscopy differed considerably. Figures 21 and 22 show structures typical of the simple 1/2 h/Z.A. temperature/A.C. heat treatment prior to rupture testing. Both Alloys 44 and 49 showed well defined cuboidal primary γ' with some in-fill of finer secondary γ' that formed during air cooling. The primary γ' size was approximately 1-2 μm and occupied a volume fraction of approximately 70%. The effect of B/Zr additions to Alloy 44 (Figure 21) was apparent as an increase in grain boundary γ' precipitation, as shown in Figure 21 of Alloy 49. The more contiguous nature of the grain boundaries in this alloy may account for the increase in rupture strength/ductility over Alloy 44. Similar effects have been observed elsewhere(13).

Figures 23 and 24 show that the combination heat treatment of 2 h/2360°F (1295°C)/FAC + 24 h/1660°F (905°C)/ A.C. on Alloys 44 and 49, respectively, produced markedly different structures than the simple 1/2 h/Z.A. temperature/ A.C. heat treatment. The B/Zr modified Alloy 49 still showed an improved rupture life over Alloy 44, but the γ' morphology of both alloys was complex and the micrographs indicated the presence of another or other precipitates (light etching). These were subsequently identified as carbides (see Section 3.2.7). The presence of σ phase was considered unlikely, as the heat treatments improved alloy ductility, rather than produced embrittling effects associated with the presence of σ-phase.

The net effect of the combination heat treatments was to generally decrease the stress rupture life of the alloys with a moderate increase in ductility. Irrespective of which heat treatment was applied, the stress rupture properties generally increased in the order: Alloys 38, 47, 44 and 49, as shown in Table XIV. This corresponded to the same order displayed in Figures 16 and 17. Although the combination heat treatment schedules (A, B and C) gave lower rupture lives than the simple 1/2 h/Z.A. temperature/A.C. heat treatment, the 2 h/2360°F (1295°C)/FAC + 24 h/1660°F (905°C)/AC (i.e., A+C) schedule consistently gave the optimum combination of life and ductility. This schedule was intended to produce an increased volume fraction of fine γ' precipitates rather than duplex coarse and fine volume fractions that would be expected from the A+B+C schedule.

3.2.7. Structural Analyses. X-ray diffraction studies on extracted residues of alloys 44 and 49 were performed to identify the phases present after various heat treatment schedules. From phase diagram considerations(10) and alloying design the alloys were intentionally located in the γ/γ' region. However, it was feasible that the composition locus points of Alloys 44/49 could pass through say, a $\beta + \gamma + \gamma'$ phase field on cooling below the γ' solvus temperature. Whereas the cooling rate of the simple 1/2 h/2370-2390°F (1300-1310°C)/AC heat treatment appeared fast enough to suppress, say, β(NiAl) precipitation, it was possible that extended exposure in the approximate temperature range 1660-2390°F (905-1310°C) could allow this or other phases to precipitate. Selective extraction techniques (Ref. Section 2.8) were therefore used to check for the presence of β, α'(Cr), σ, carbides/carbonitrides and borides. A summary of the results is presented in Table XV which indicates that β, α'(Cr) and σ were not detected. Instead, a range of carbides were identified in a γ/γ' matrix, thus confirming the intended alloy design. It is known(14) that MC, M_6C and $M_{23}C_6$ are pseudoequilibrium phases in the 1800°F (980°C) region of compositions such as Alloy 49. Specifically, high Nb and Ta favor MC, high Mo and W favor M_6C and high Cr favors $M_{23}C_6$. As shown in Table XV, all three carbides and M_2B_3 were identified in Alloy 49. Electron microprobe analyses (Figure 25) identified Ta/Nb-rich grain boundary MC carbides and W/Mo-rich grain boundary and matrix carbides which were most likely M_6C and/or $M_{23}C_6$.

A more detailed thermal analysis was run on Alloy 49 using metallographic and Differential Thermal Analysis (DTA) techniques to characterize the phase transformation temperatures, particularly in the region of the γ' solvus temperature. The DTA results were complemented by metallographic studies on alloy samples that had been heat

treated for 2-8 hours in the temperature range 2360°C (1293°C) - 2390°F (1310°C), followed by water quenching. Figure 26 illustrates that the γ' solvus temperature of Alloy 49 was approximately 2320°F (1270°C). Significant resolutioning of the γ' did not occur until 2390°F (1310°C) when evidence of incipient melting at grain boundary triple points was observed. The residual high volume fraction of γ' precipitation at 2360°F (1293°C) thereby lowered the effectiveness of the 2 h/2360°F (1293°C)/Fast AC solution treatment investigated in Section 3.2.6. This could account for the lower rupture properties obtained with the combination heat treatments than with the simple 1/2 h/Z.A. temperature/AC treatments which gave better γ' distribution and morphology (compare Figures 22 and 24).

The other significant phase changes indicated by the DTA trace occurred in the mushy zone of the alloy. The pronounced peaks at 2510°F (1377°C) and 2540°F (1393°C) were considered to be due to MC carbide solution (for example, Ta/Nb-rich and W-rich MC, respectively).

3.3 General Discussion

The characterization of the three best alloys has essentially been completed. The results confirmed that the high volume % γ' content, characteristic of these alloys, is an effective means of improving the intermediate temperature strength of ODS superalloys. Extending the program schedule to evaluate minor additions of B and Zr in these alloys showed that the 1400°F (760°C)/100-hour rupture strength was further improved to 90 ksi (620.5 MPa) in Alloy 49 with a 2000°F (1093°C)/100-hour rupture strength of 19.5 ksi (134 MPa).. It was not possible to complete hot corrosion studies on Alloys 49-51 in the time available, mainly because of the additional B/Zr development work. However, extensive hot corrosion data determined in the previous contract(6) on similar Ni-Cr-Al-W/Mo/Ta/Nb quaternary alloys indicated that Alloys 49-51 should display better oxidation resistance than IN-100 and sulfidation resistance much superior to IN-713C, IN-100 or B-1900. A major goal of this research contract has thereby been achieved: namely, deriving the combination of excellent intermediate temperature strength through high γ' content, plus outstanding high temperature strength by virtue of the oxide dispersion and directional grain shape in a corrosion resistant alloy.

During the gradient anneal studies, an attempt was made to relate the secondary recrystallization and solvus temperatures of the experimental alloys. It was apparent that the initiation of secondary recrystallization occurred at or above the γ' solvus temperature. This implies that a proportion of the γ' must redissolve before

growth of the recrystallized grains can proceed, and that the redissolution rate is, in turn, proportional to the γ' volume fraction of the alloy. The temperature range in which secondary recrystallization can occur in these alloys can, therefore, be extended, inter alia, by lowering the γ' solvus temperature.

Since the γ' solvus temperature and volume fraction are both functions of alloy composition, it is possible to extend the temperature range of directional recrystallization response in a given alloy by minor compositional modifications. This effect is evident on comparing the recrystallization response of Alloys 49 (Figure 12) and 51 (Figure 14). Thus a composition modification may be desirable in Alloy 49; the best 1400°F (760°C) strength alloy developed to date.

A slight reduction in the content of γ'-forming elements such as Al and Nb, coupled with a minor increase in the Mo content (known(6) to favor directional grain growth), should extend the temperatue range of directional recrystallization response by the mechanism discussed above. An alternative approach is to modify Alloy 51 which showed the best directional recrystallization response and 2000°F (1093°C) strength (Section 3.2.5.1), through minor additions of Ta and Nb, to improve the 1400°F (760°C) strength, without raising the γ' solvus temperature significantly. Both alloy modifications represent optimization approaches aimed ultimately at a single alloy composition. The two compositions (in w/o) are identified as:

A: Ni-11Cr-6Al-6.5W-3Mo-3Ta-1Nb-0.15Zr-0.01B-1.1Y_2O_3

- and -

B: Ni-9.5Cr-8Al-6.5W-3Mo-1Ta-1Nb-0.15Zr-0.01B-1.1Y_2O_3

Detailed characterization of the final high volume % γ' alloy can be addressed once these optimization studies have been completed.

IV. CONCLUSIONS

1. Development of a series of high volume % γ' nickel-base ODS superalloys confirmed that the high % γ' characteristic is an effective means of improving the intermediate temperature strength. An alloy having 100-hour rupture strengths of 90 ksi (620 MPa) at 1400°F (760°C) and 19.5 ksi (134 MPa) at 2000°F (1093°C), respectively, has been identified.

2. Extensive hot corrosion data(6) indicated that the compositional range of the series has excellent oxidation resistance (generally better than IN-100 and much superior to IN-713 and IN-738) and much superior sulfidation resistance to IN-100, IN-713 and B-1900.

3. Directional recrystallization response was improved by secondary hot working. Maximum strength was developed with a simple heat treatment.

4. Correlation between the γ' content, γ' solvus temperature and directional recrystallization temperature of the series suggested minor compositional changes to optimize the properties of a single alloy.

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TABLE I

SERIES III ALLOYS (COMPOSITIONS IN ATOMIC AND WEIGHT %)

Alloy No.	Batch No.	Atomic %*							Weight %							
		Ni	Cr	Al	W	Mo	Ta	Nb	Ni	Cr	Al	W	Mo	Ta	Nb	Y_2O_3
37	V121, V123	67	15	15	2	-	1	-	68.3	13.8	7.1	6.5	-	3.2	-	1.1
38	V126	66	15	15.5	2	1	0.5	-	67.8	13.9	7.4	6.5	1.7	1.6	-	1.1
39	V129	65	15	16	2	2	-	-	67.2	14.0	7.7	6.6	3.4	-	-	1.1
40	V132	66	15	14	2	1	1	1	66.1	13.5	6.5	6.4	1.7	3.1	1.6	1.1
41	V135	66	12.5	16.5	2	-	1	-	69.0	11.8	8.1	6.7	-	3.3	-	1.1
42	V138	67	12.5	17	2	1	0.5	-	69.2	11.6	8.2	6.6	1.7	1.6	-	1.1
43	V141	66	12.5	17.5	2	2	-	-	68.6	11.7	8.5	6.6	3.5	-	-	1.1
44	V144	67	12.5	15.5	2	1	1	1	67.5	11.3	7.3	6.4	1.7	3.2	1.6	1.1
45	V147	70.5	10	16.5	2	-	1	-	72.1	9.2	7.9	6.5	-	3.2	-	1.1
46	V150	69.5	10	17	2	1	0.5	-	71.5	9.3	8.2	6.6	1.7	1.6	-	1.1
47	V153	68.5	10	17.5	2	2	-	-	71.1	9.3	8.5	6.6	3.4	-	-	1.1
48	V156	69.5	10	15.5	2	1	1	1	69.8	9.0	7.3	6.4	1.7	3.1	1.6	1.1

Alloys 37-40 based on Ni-15 At.%Cr-16 At.% Al ternary
Alloys 41-44 based on Ni-12.5 At.%Cr-17.5 At.% Al ternary
Alloys 45-48 based on Ni-10 At.%Cr-17.5 At.% Al ternary

*Excluding added Y_2O_3

TABLE II

POWDER CHARACTERIZATION OF SERIES III ALLOYS

Alloy No.	Batch No.	Chemistry (Wt.%)			Screen Analysis, Mesh Size (%)						
		O	N	C	+40	-40/+80	-80/+100	-100/+140	-140/+200	-200/+325	-325
37+	V121*	.16	.055	.046	17.9	31.8	6.9	11.4	13.8	9.6	8.6
37+	V122	.10	.058	.045	29.7	30.7	5.7	8.5	10.1	8.7	6.6
37	V123	.19	.059	.047	15.6	33.7	9.6	12.4	12.2	8.7	7.8
37	V124	.16	.057	.045	13.3	36.8	9.3	11.8	12.8	8.3	7.7
38	V126	.32	.13	.046	13.1	40.4	8.5	11.8	11.5	7.3	7.4
38	V127	.16	.044	.044	9.0	37.2	8.7	11.9	12.6	10.1	10.5
39	V129	.12	.043	.042	15.7	38.3	9.2	11.2	10.5	7.9	7.2
39	V130	.23	.051	.042	19.3	41.8	7.4	9.7	9.2	6.4	6.2
40	V132	.16	.057	.042	25.8	37.8	7.2	9.0	8.3	6.5	5.4
40	V133	.24	.054	.045	21.0	40.8	7.5	8.4	8.2	6.7	7.4
41	V135	.55	.062	.045	11.5	42.6	10.2	11.0	10.2	6.7	7.8
41	V136	.50	.058	.044	20.7	42.4	7.6	9.1	8.3	6.0	5.9
42	V138	.16	.060	.045	21.9	37.8	8.5	9.9	9.1	6.2	6.6
42	V139	.16	.062	.045	14.0	38.3	8.4	11.0	11.2	9.0	8.1
43	V141	.29	.058	.042	17.2	36.7	10.5	10.8	9.9	8.0	6.9
43	V142	.42	.059	.042	18.4	44.4	8.3	9.3	7.9	5.9	5.8
44	V144	.29	.062	.044	11.9	39.8	9.5	11.9	10.9	8.5	7.5
44	V145	.42	.057	.043	13.7	35.2	8.6	12.0	11.7	9.2	9.6
45	V147	.16	.050	.045	15.4	34.0	9.0	11.4	12.5	9.2	8.5
45	V148	.20	.050	.045	20.9	40.7	8.3	10.2	9.1	6.0	4.8
46	V150	.43	.053	.044	14.6	41.8	9.4	10.1	10.1	7.2	6.8
46	V151	.18	.054	.044	26.2	38.0	8.3	9.7	7.7	5.5	4.5
47	V153	.14	.042	.047	20.0	42.7	7.6	9.3	8.0	5.9	6.5
47	V154	.16	.040	.044	15.9	49.7	6.9	9.4	8.1	5.6	4.4
48	V156	.28	.047	.048	17.1	43.3	9.2	10.4	9.3	6.4	4.2
48	V157	.31	.042	.046	14.7	33.0	7.7	11.5	12.7	10.6	9.8

+Extruded in double-walled extrusion cans. All remaining batches extruded in single-walled extrusion cans.

*The batch number V121 was assigned to the total of the combined Alloy 37 batches V121 and V122 which were cone blended. Likewise, V126, V129, etc. were assigned to the two cone blended batches of Alloys 38, 39, etc. respectively.

TABLE III

EXTRUSION CONDITIONS OF SERIES III ALLOYS

Alloy No.	Billet No.	Temperature °F	Temperature (°C)	Preheat Time (h)	Ratio	Ram Speed* in/sec	Ram Speed* (cm/sec)
37	V121A	1850	(1010)	1	18:1	3.4	8.6
	V121B	1950	(1065)	1	18:1	3.6	9.1
	V121C	2050	(1120)	1	18:1	4.0	10.1
	V121D	2050	(1120)	2	18:1	3.6	9.1
	V123A	1850	(1010)	1	18:1	1.6	4.1
	V123B	1950	(1065)	1	18:1	2.0	5.1
	V123C	2150	(1177)	1	18:1	2.0	5.1
38	V126A	1850	(1010)	2	18:1	0.4	1.0
	V126B	1950	(1065)	1 1/2	18:1	2.0	5.1
	V126C	2050	(1120)	1	18:1	---No Record---	
	V126D	2150	(1177)	1	18:1	3.6	9.1
39	V129A	1850	(1010)	2	18:1	4.0	10.1
	V129B	1950	(1065)	1 1/2	18:1	2.1	5.3
	V129C	2050	(1120)	1	18:1	2.8	7.1
	V129D	2150	(1177)	1	18:1	3.6	9.1
40	V132A	1850	(1010)	2	18:1	0.8	2.0
	V132B	1950	(1065)	1 1/2	18:1	2.1	5.3
	V132C	2050	(1120)	1	18:1	2.8	7.1
	V132D	2150	(1177)	1	18:1	4.0	10.1
41	V135A	1850	(1010)	2	18:1	2.0	5.1
	V135B	1950	(1065)	1 1/2	18:1	2.8	7.1
	V135C	2050	(1120)	1	18:1	3.2	8.1
42	V138A	1850	(1010)	2	18:1	1.6	4.1
	V138B	1950	(1065)	1 1/2	18:1	2.4	6.1
	V138C	2050	(1120)	1	18:1	3.2	8.1
	V138D	2150	(1177)	1	18:1	4.0	10.1
43	V141A	1850	(1010)	2	18:1	1.6	4.1
	V141B	1950	(1065)	1 1/2	18:1	2.8	7.1
	V141C	2050	(1120)	1	18:1	3.2	8.1
	V141D	2150	(1177)	1	18:1	---No Record---	
44	V144A	1850	(1010)	2	18:1	4.0	10.2
	V144B	1950	(1065)	1 1/2	18:1	5.6	14.2
	V144C	2050	(1120)	1	18:1	6.0	15.2
	V144D	2150	(1177)	1	18:1	4.0	10.1
45	V147A	1950	(1065)	1 1/2	18:1	3.4	8.6
	V147B	2050	(1120)	1	18:1	3.4	8.6
	V147C	2150	(1177)	1	18:1	3.6	9.1
46	V150A	1950	(1065)	1 1/2	18:1	3.0	7.6
	V150B	2050	(1120)	1	18:1	3.8	9.6
	V150C	2150	(1177)	1	18:1	4.0	10.1
47	V153A	1950	(1065)	1 1/2	18:1	1.6	4.1
	V153B	2050	(1120)	1	18:1	2.4	6.1
	V153C	2150	(1177)	1	18:1	2.4	6.1
48	V156A	1950	(1065)	1 1/2	18:1	1.8	4.6
	V156B	2050	(1120)	1	18:1	2.0	5.1
	V156C	2150	(1177)	1	18:1	2.4	6.1

*35% press throttle

TABLE IV

PRELIMINARY STRESS RUPTURE RESULTS (STEP LOAD)

Alloy No.	Bar No.	Zone Anneal Temperature* °F	(°C)	Zone Anneal Speed iph	(cmph)	Temperature °F	(°C)	Stress ksi	(MPa)	Life (h)	Elong. (%)	R.A. (%)
38	V126D	2360	(1293)	2.8	(7.1)	2000	(1093)	14	(96.5)	24.0	Step	Load
								16	(110.0)	24.0	Step	Load
								18	(124.0)	0.1	Nil	Nil
42	V138D	2360	(1293)	2.8	(7.1)	2000	(1093)	14	(96.5)	24.0	Step	Load
								16	(110.0)	16.9	1.3	2.9
44	V144D	2360	(1293)	2.8	(7.1)	2000	(1093)	14	(96.5)	24.0	Step	Load
								16	(110.0)	24.0	Step	Load
								18	(124.0)	24.0	Step	Load
								20	(138.0)	15.8	2.5	2.9
47	V153C	2360	(1293)	2.8	(7.1)	2000	(1093)	14	(96.5)	24.0	Step	Load
								16	(110.0)	24.0	Step	Load
								18	(124.0)	24.0	Step	Load
								20	(138.0)	9.2	2.5	Nil

* Post zone anneal heat treatment given: 1/2 h/Z.A. temperature/AC

TABLE V

STRESS RUPTURE RESULTS

Alloy No.	Bar No.	Zone Anneal Temperature* (°F)	(°C)	Zone Anneal Speed iph	(cmph)	Temperature °F	(°C)	Stress ksi	(MPa)	Life (h)	El. (%)	R.A. (%)
38	V126D	2360	(1293)	2.8	(7.1)	2000	(1093)	18	(124)	2.9	2.5	1.5
		"	"	"	"	"	"	18	(124)	2.5	1.3	Nil
		"	"	"	"	"	"	16	(110)	24.0	1.0	"
		"	"	"	"	"	"	16	(110)	16.3	1.3	"
		2390	(1310)	"	"	1400	(760)	100	(689.5)	9.5	1.3	4.7
		"	"	"	"	"	"	90	(620.5)	31.9	2.5	4.7
		2360	(1293)	"	"	"	"	80	(552)	80.2	Nil	Nil
		"	"	"	"	"	"	70	(483)	535.8	2.5	"
44	V144D	"	"	"	"	2000	(1093)	20	(138)	14.0	2.5	2.9
		"	"	"	"	"	"	20	(138)	15.8	2.5	2.9
		"	"	"	"	"	"	18	(124)	28.5	1.3	Nil
		"	"	"	"	"	"	18	(124)	40.0	2.5	2.9
		"	"	"	"	1400	(760)	100	(689.5)	7.3	1.3	6.7
		2390	(1310)	"	"	"	"	90	(620.5)	24.9	1.3	4.1
		2360	(1293)	"	"	"	"	85	(586)	94.3	1.3	1.5
		"	"	"	"	"	"	75	(517)	368.6	2.5	Nil
47	V153C	"	"	"	"	2000	(1093)	20	(138)	9.2	2.5	Nil
		"	"	"	"	"	"	20	(138)	8.2	Nil	"
		"	"	"	"	"	"	18	(124)	29.1	"	"
		"	"	"	"	"	"	18	(124)	33.2	2.5	"
		"	"	"	"	1400	(760)	100	(689.5)	7.2	1.3	1.5
		2390	(1310)	"	"	"	"	90	(620.5)	23.8	1.4	4.7
		2360	(1293)	"	"	"	"	85	(586)	67.8	1.3	1.5
		"	"	"	"	"	"	75	(517)	159.1	1.3	2.9
		"	"	"	"	"	"	70	(483)	371.7	1.3	6.0

* Post zone anneal heat treatment given: 1/2 h/Z.A. temperature/AC.

TABLE VI

EXPERIMENTAL ALLOY COMPOSITIONS (IN ATOMIC AND WEIGHT %)

Alloy No.	Batch No.	Atomic,%*									Weight,%									
		Ni	Cr	Al	W	Mo	Ta	Nb	Zr	B	Ni	Cr	Al	W	Mo	Ta	Nb	Zr	B	Y_2O_3
38	V126	66	15	15.5	2	1	0.5	-	-	-	67.8	13.9	7.4	6.5	1.7	1.6	-	-	-	1.1
44	V144	67	12.5	15.5	2	1	1	1	-	-	67.4	11.3	7.3	6.4	1.7	3.2	1.6	-	-	1.1
47	V153	68.5	10	17.5	2	2	-	-	-	-	71.1	9.3	8.5	6.6	3.4	-	-	-	-	1.1
49	{V159 V163	66.8	12.5	15.5	2	1	1	1	0.09	0.05	68.3	11.3	7.3	6.4	1.7	3.2	1.6	0.15	0.01	1.1
50	V167	65.8	15	15.5	2	1	0.5	-	0.09	0.05	68.7	13.9	7.4	6.5	1.7	1.6	-	0.15	0.01	1.1
51	V170	70.7	10	17.5	2	2	-	-	0.09	0.05	70.9	9.3	8.5	6.6	3.4	-	-	0.15	0.01	1.1

* Excluding added Y_2O_3.

TABLE VII

POWDER CHARACTERIZATION OF EXPERIMENTAL ALLOYS CONTAINING B AND Zr

No.	Batch	Chemistry (wt.%)			Screen Analysis, Mesh Size (%)						
		O	N	C	+40	-40/+80	-80/+100	-100/+140	-140/+200	-200/+325	-325
49	V-159*	.14	.060	.062	20.1	34.6	5.6	8.6	10.3	10.3	10.5
49	V-160	.20	.060	.050	12.4	30.1	7.5	11.8	12.7	12.5	13.0
49	V-161	.21	.058	.067	13.8	33.9	7.3	11.3	11.5	11.3	10.9
49	V-162	.26	.053	.080	5.5	27.2	7.3	12.5	20.0	10.6	16.9
49	V-163	.25	.10	.053	12.2	31.4	7.9	11.0	13.4	9.9	14.2
49	V-164	.26	.081	.051	18.2	31.8	6.2	10.1	11.7	10.2	11.8
49	V-165	.43	.085	.051	13.7	27.2	6.4	11.9	13.3	13.2	14.3
50	V-167	.65	.063	.047	14.5	32.3	7.4	11.8	11.9	10.5	11.6
50	V-168	.37	.069	.049	17.3	39.8	8.6	10.6	9.1	7.6	7.0
51	V-170	.24	.055	.047	10.5	30.8	9.3	14.0	13.5	11.3	10.6
51	V-171	.35	.055	.050	20.9	35.1	8.4	10.0	9.9	8.0	7.7

* Batch No. V-159 was assigned to the total of the combined Alloy 49 batches, V-159. V-160, V-161 and V-162 which were cone blended. Batch No. V-163 was assigned to the total of the combined Alloy 49 batches, V-163, V-164 and V-165 which were cone blended. Likewise, V-167 and V-170 were assigned to the cone blended batches of Alloys 50 and 51, respectively.

TABLE VIII

EXTRUSION CONDITIONS OF EXPERIMENTAL ALLOYS CONTAINING B AND Zr

Alloy No.	Billet No.	Temperature °F	Temperature (°C)	Preheat Time (h)	Ratio	Ram Speed* in/sec	Ram Speed* (cm/sec)
49	V159A	1850	(1010)	2	18:1	3.0	7.6
	V159B	1950	(1065)	1-1/2	18:1	3.8	9.6
	V159C	2020	(1120)	1	18:1	4.0	10.2
	V159D	2150	(1177)	1	18:1	4.2	10.7
	V159E	2200	(1204)	1	18:1	4.2	10.7
49	V-163A	2150	1175	1	18:1	3.6	9.1
	V-163B	2150	1175	1	10:1	4.4	11.2
	V-163C	2100	1150	1-1/2	18:1	2.4	6.1
	V-163D	2100	1150	1-1/2	10:1	4.2	10.7
	V-163E	2150	1175	1-1/2	18:1	3.8	9.6
	V-163F	2150	1175	1-1/2	10:1	4.2	10.7
	V-163G	2200	1205	1-1/2	18:1	4.0	10.2
50	V-163H	2200	1205	1-1/2	10:1	4.4	11.2
	V-167A	2150	1175	1-1/2	18:1	2.7	6.8
	V-167B	2150	1175	1-1/2	10:1	4.4	11.2
	V-167C	2150	1175	1	18:1	4.0	10.2
51	V-167D	2150	1175	1	10:1	4.3	10.9
	V-170A	2150	1175	1	18:1	4.2	10.7
	V-170B	2150	1175	1	10:1	4.3	10.9
	V-170C	2150	1175	1-1/2	18:1	4.0	10.2
	V-170D	2150	1175	1-1/2	10:1	4.4	11.2

* 35% Press Throttle.

TABLE IX

HOT ROLLING CONDITIONS FOR ALLOYS 49-51

Hot Roll Temp. (°F)	Hot Roll Temp. (°C)	Hot Roll Thickness Reduction (%) 20	40	60
2250	(1230)	V163D-1	V163D-2 V167D-1 V170D-1	V163D-3
2150	(1175)	V163F-1 V167B-1 V170B-1	V163F-2 V167B-2 V167D-2 V170B-2 V170D-2	V163F-3 V167B-3 V170B-3
2050	(1120)	V163B-1	V163B-2 V167D-3 V170D-3	V163B-3

NOTE: Bars preheated 1 hour at temperature, thickness reduced 20% per pass with 10-15 min reheats at temperature between passes.

Alloy 49 = Bars V163 etc.
Alloy 50 = Bars V167
Alloy 51 = Bars V170

TABLE X

STRESS RUPTURE RESULTS OF B/Zr MODIFIED ALLOYS

Alloy No.	Bar No.	Zone Anneal Temperature* (°F)	(°C)	Zone Anneal Speed iph	(cmph)	Temperature (°F)	(°C)	Stress ksi	(MPa)	Life (h)	El. (%)	R.A. (%)	Density lb/in³	(g/cc)
49	V159D	2370	(1299)	2.8	7.1	2000	(1093)	20	(138)	20.4	Nil	3.3	0.289	(8.01)
		"	"	"	"	"	"	20	(138)	14.8	1.3	2.7		
		"	"	"	"	"	"	20	(138)	29.6	Nil	2.8		
		"	"	"	"	1400	(760)	100	(689.5)	19.5	1.3	4.7		
		"	"	"	"	"	"	90	(620.5)	97.3	1.3	4.7		
		"	"	"	"	"	"	85	(586)	181.9	1.3	3.4		
		"	"	"	"	"	"	80	(552)	413.6	1.3	6.1		
50	V167A	2330	(1277)	2.8	7.1	2000	(1093)	20	(138)	3.2	2.5	4.1	0.289	(8.01)
		"	"	"	"	"	"	20	(138)	2.4	2.5	7.3		
		"	"	"	"	1400	(760)	85	(586)	68.2	1.3	2.7		
		"	"	"	"	"	"	85	(586)	115.4	1.3	3.3		
51	V170A	2330	(1277)	2.8	7.1	2000	(1093)	20	(138)	27.9	3.8	3.4	0.286	(7.93)
		"	"	"	"	"	"	20	(138)	53.5	1.3	1.4		
		"	"	"	"	1400	(760)	85	(586)	106.7	1.3	2.8		
		"	"	"	"	"	"	85	(586)	127.6	2.5	2.8		

* Post zone anneal heat treatment given: 1/2 h/Z.A. temperature/AC.

TABLE XI

EXPERIMENTAL ALLOY RUPTURE STRENGTHS

Alloy No.	Bar No.	Temperature °F	Temperature (°C)	Strength, ksi(MPa) 100-Hour		Strength, ksi(MPa) 1000-Hour(1)	
38	V126D[2]	1400	(760)	80	(552)	66	(455)
		2000	(1093)	15	(103)	13	(90)
44	V144D[2]	1400	(760)	85	(586)	70	(483)
		2000	(1093)	19	(131)	18	(124)
47	V153C[2]	1400	(760)	81	(558)	67	(462)
		2000	(1093)	18.5	(127.5)	16	(110)
49	V159D[2]	1400	(760)	90	(620.5)	76	(524)
		2000	(1093)	19.5	(134)	19	(131)
MA 6000E[3]		1400	(760)	80	(552)	70	(483)
		2000	(1093)	23	(159)	22	(152)
IN-100[4]		1400	(760)	91	(627)	75	(517)
		2000	(1093)	9	(62)	2	(14)
DS MAR-M200 + Hf[5]		1400	(760)	105	(724)	90	(620.5)
		2000	(1093)	10	(69)	5	(34)

NOTES: (1) Strength levels at 1000-hour test duration are estimated values.

(2) Zone annealed and heat treated 1/2 h/Z.A. temperature/A.C.

(3) Composition (wt.%): Ni-15Cr-4.5Al-4W-2Mo-2.5Ti-2Ta-0.15Zr-0.01B-1.1Y_2O_3, zone annealed and heat treated 2250°F(1230°C)/1/2 h/AC + 1750°F(955°C)/ 2 h/AC + 1550°F(845°C)/24 h/AC.

(4) As-Cast

(5) From Ref. 11 - fully heat treated.

TABLE XII

100-HOUR RUPTURE STRENGTH DATA FOR FIGURE 18

Alloy	Density		Temperature		Stress, σ		σ/ρ	
	ρ, lb/in^3	(g/cm^3)	(°F)	(°C)	(ksi)	(MPa)	in x 10^3	(cm x 10^3)
Alloy 49	0.289	(8.01)	1400	(760)	90	(620.5)	311	(775)
			2000	(1093)	19.5	(134)	67.5	(167)
MA 6000E	0.293	(8.11)	1400	(760)	80	(552)	273	(681)
			2000	(1093)	23	(159)	78.5	(196)
IN-100	0.280	(7.75)	1400	(760)	91	(627)	325	(809)
			2000	(1093)	9	(62)	32	(80)
DS MAR-M200 + Hf	0.312	(8.63)	1400	(760)	105	(724)	336	(838)
			2000	(1093)	10	(69)	29	(79)
DS Eutectic $\gamma/\gamma'+\alpha$*	0.307	(8.5)	1400	(760)	125	(862)	407	(1014)
			2000	(1093)	19	(131)	62	(154)

* From Reference 11.

TABLE XIII

TENSILE TEST RESULTS

Alloy No.	Bar No.	Tensile Test Temperature °F	(°C)	0.2% PS ksi	(MPa)	U.T.S. ksi	(MPa)	El. (%)	R.A. (%)	Modulus psix10^6	(MPax10^3)
38	V126D(1)	RT		143.5	(989)	144.9	(999)	3.5	9.0	31.1	(214.4)
		1400	(760)	138.8	(957)	148.0	(1020)	2.0	3.5	9.1	(62.7)
44	V144D(1)	RT		161.1	(1111)	173.6	(1197)	3.5	11.0	32.3	(222.7)
		1400	(760)	166.7	(1149)	166.7	(1149)	2.0	2.5	9.7	(66.9)
47	V153C(1)	RT		147.9	(1020)	173.5	(1196)	7.0	8.5	31.3	(215.8)
		1400	(760)	145.6	(1004)	145.9	(1006)	2.0	5.5	11.9	(82.1)
49	V159D(2)	RT		169.4	(1168)	183.3	(1264)	3.5	8.0	34.7	(239.2)
		1400	(760)	166.7	(1149)	166.7	(1149)	1.0	2.5	12.8	(88.2)
50	V167A(3)	RT		157.8	(1088)	176.2	(1215)	3.5	8.5	35.4	(244.1)
		1400	(760)	152.7	(1053)	152.7	(1053)	2.0	3.0	10.6	(73.1)
51	V170A(3)	RT		150.0	(1034)	169.9	(1171)	2.0	3.5	31.7	(218.6)
		1400	(760)	151.4	(1044)	160.1	(1107)	3.5	6.5	12.3	(84.8)
IN-100(4)		RT		123	(850)	147	(1018)	9	-	31.2	(215.1)
		1400	(760)	125	(860)	155	(1070)	6.5	-	25.1	(173.1)
DS MAR-M-200 + Hf(5)		RT		126	(869)	158	(1089)	13.1	16.7	19.4	(133.7)
		1400	(760)	131.5	(907)	166	(1145)	11.7	22.8	13.2	(91.0)

NOTES: (1) Zone annealed at 2390°F(1310°C)/2.8 iph(7.1 cmph) and heat treated 1/2 h/2390°F(1310°C)/AC.

(2) Zone annealed at 2340°F(1280°C)/2.8 iph(7.1 cmph) and heat treated 1/2 h/2340°F(1280°C)/AC.

(3) Zone annealed at 2330°F(1280°C)/2.8 iph(7.1 cmph) and heat treated 1/2 h/2330°F(1280°C)/AC.

(4) As-Cast.

(5) From Ref. 12 - fully heat treated.

TABLE XIV

EFFECT OF HEAT TREATMENT ON STRESS RUPTURE PROPERTIES OF ALLOYS 38, 44, 47 AND 49

Alloy No.	Bar No.	Zone Anneal Temperature* (°F)	(°C)	Heat Treatment**	Temperature (°F)	(°C)	Stress ksi	(MPa)	Life (h)	El. (%)	R.A. (%)
38	V126D	2390	(1310)	A+B+C	1400	(760)	85	(586)	42.9	1.3	6.6
"	"	"	"	A+B	"	"	"	"	57.7	1.3	3.4
"	"	"	"	A+C	"	"	"	"	59.2	2.5	4.7
"	"	"	"	1/2 h/ZA/AC	"	"	"	"	48.1	1.5	2.5
"	"	"	"	A+B+C	2000	(1093)	20	(138)	2.0	3.8	12.1
"	"	"	"	A+B	"	"	"	"	1.7	1.3	12.8
"	"	"	"	A+C	"	"	"	"	1.5	3.8	12.5
"	"	"	"	A+C	"	"	"	"	0.5	2.8	3.0
44	V144D	2390	(1310)	A+B+C	1400	(760)	85	(586)	34.9	1.3	4.8
"	"	"	"	A+B	"	"	"	"	75.8	1.3	5.9
"	"	"	"	A+C	"	"	"	"	65.2	2.5	7.2
"	"	"	"	1/2 h/ZA/AC	"	"	"	"	94.3	1.3	1.5
"	"	"	"	A+B+C	2000	(1093)	20	(138)	2.2	1.3	2.7
"	"	"	"	A+B	"	"	"	"	9.1	1.3	3.3
"	"	"	"	A+C	"	"	"	"	8.8	1.3	4.7
"	"	"	"	1/2 h/ZA/AC	"	"	"	"	14.0	2.5	2.9
47	V153C	2390	(1310)	A+B+C	1400	(760)	85	(586)	27.8	1.3	4.6
"	"	"	"	A+B	"	"	"	"	28.0	2.5	4.7
"	"	"	"	A+C	"	"	"	"	28.5	1.3	6.8
"	"	"	"	1/2 h/ZA/AC	"	"	"	"	67.8	1.3	1.5
"	"	"	"	A+B+C	2000	(1093)	20	(138)	12.5	2.5	5.4
"	"	"	"	A+B	"	"	"	"	18.5	1.3	3.3
"	"	"	"	A+C	"	"	"	"	4.0	2.5	4.0
"	"	"	"	1/2 h/ZA/AC	"	"	"	"	9.2	2.5	Nil
49	V159D	2370	(1300)	A+B+C	1400	(760)	85	(586)	107.2	3.8	2.6
"	"	"	"	A+B	"	"	"	"	79.3	2.5	3.3
"	"	"	"	A+C	"	"	"	"	123.8	3.8	2.6
"	"	"	"	1/2 h/ZA/AC	"	"	"	"	181.9	1.3	3.4
"	"	"	"	A+B+C	2000	(1093)	20	(138)	9.7	1.3	Nil
"	"	"	"	A+B	"	"	"	"	7.5	1.3	2.0
"	"	"	"	1/2 h/ZA/AC	"	"	"	"	14.8	1.3	2.7

* Zone anneal speed 2.8 iph(7.1 cmph).

** A = 2 h/2360°F(1295°C)/FAST AC
B = 4 h/2060°F(1130°C)/AC
C = 24 h/1660°F(905°C)/AC
1/2 h/ZA/AC = 1/2 h/Zone Anneal Temperature/AC

TABLE XV

X-RAY DIFFRACTION ANALYSIS OF ALLOYS 44 AND 49

Alloy (Bar No.)	Heat Treatment[1]	1400°(760°C)/ 85 ksi Life (h)	Extraction Method	ASTM File Card Number 6-0694 α' (Cr-rich)	2-1261 β (NiAl)	(3) γ' (Ni_3Al)	(3) MC	14-407 $M_{23}C_6$	(3) M_6C	11-550 M_7C_3	(3) σ	(3) M_3B_2
44(V144D)	A+C	65	Anodic	ND	ND	x	x	x	x	ND	ND	ND
"	A+B+C	35	Berzelius	ND	ND	ND	x	x	x	ND	ND	ND
49(V159D)	1/2 h/2370°F/AC	182	Anodic	ND	ND	x	x	x	x	ND	ND	x
"	A+B	79	"	ND	ND	x	x	x	x	ND	ND	x
"	A+C	124	"	ND	ND	x	x	x	x	ND	ND	x
"	A+B+C	107	Berzelius	ND	ND	ND	x	x	x	ND	ND	x

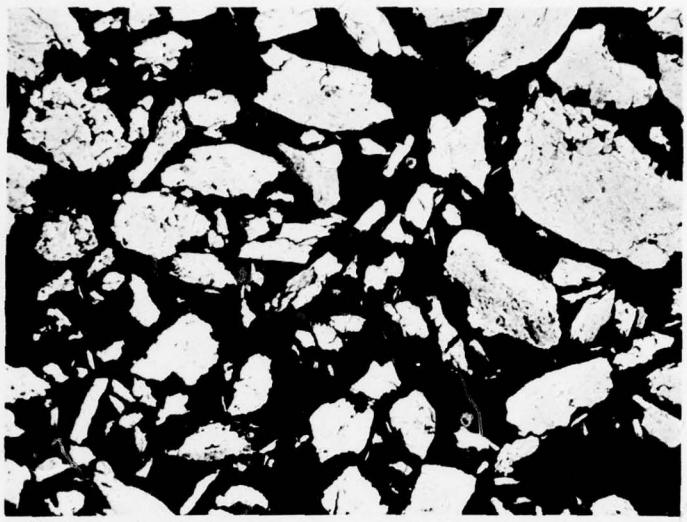
NOTES: N.D. = None Detected.

(1) Prior to heat treatment, Bar No. V144D was zone annealed 2390°F(1310°C)/2.8 iph(7.1 cmph) and Bar No. V159D was zone annealed 2370°F(1300°C)/2.8 iph(7.1 cmph).

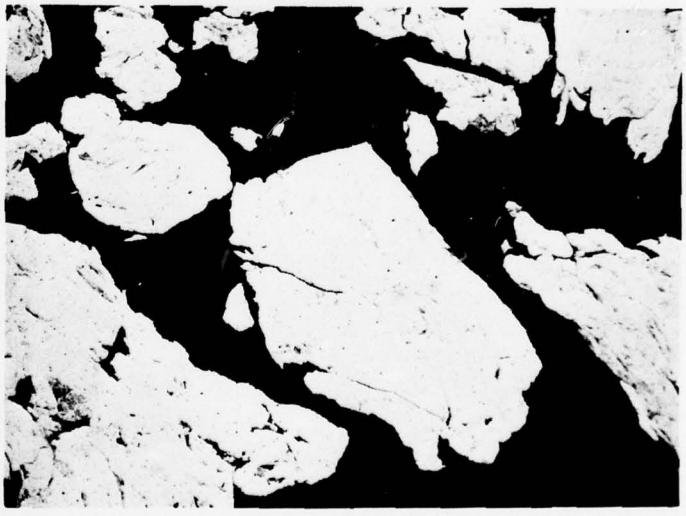
(2) Heat Treatments: A = 2 h/2360°F(1295°C)/Fast Air Cool
B = 4 h/2060°F(1130°C)/AC
C = 24 h/1660°F(905°C)/AC

(3) Ref. Donachie, M. J. and Kriege, O. H., J. Materials, JMLSA, Vol. 7, No. 3, Sept. 1972, p. 269-278.

(4) Reflections for γ and Y_2O_3/Al_2O_3 combinations (predominately as $3Y_2O_3 \cdot 5Al_2O_3$) detected in all samples.

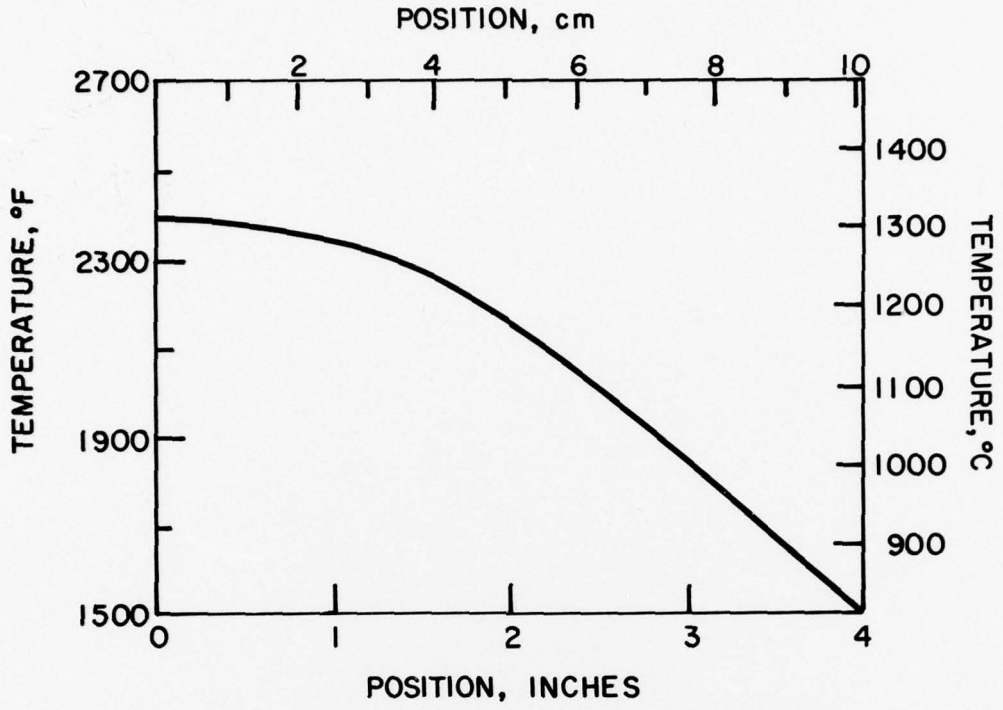


PN 1-71401 50X



PN 1-71402 250X

FIGURE 1: Micrographs of attrited powder of Alloy 42



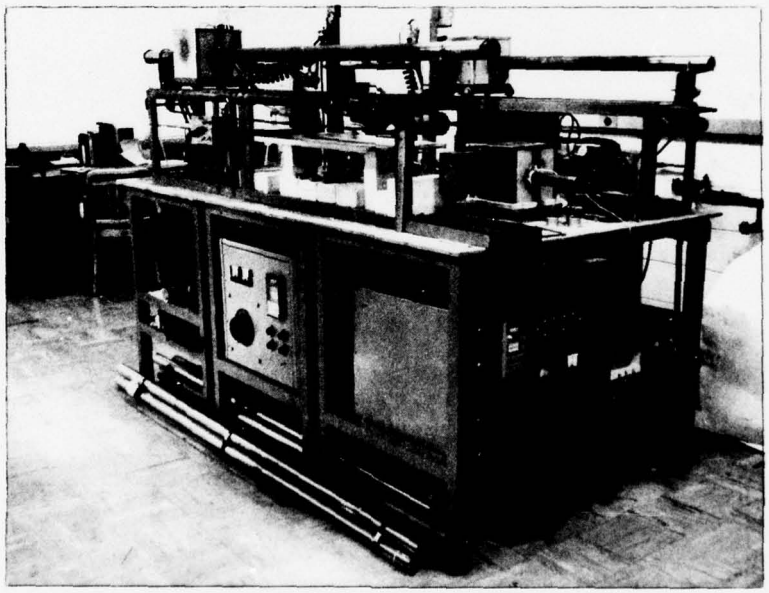


FIGURE 2 - Temperature Profile and Furnace Used for Gradient and Zone Annealing

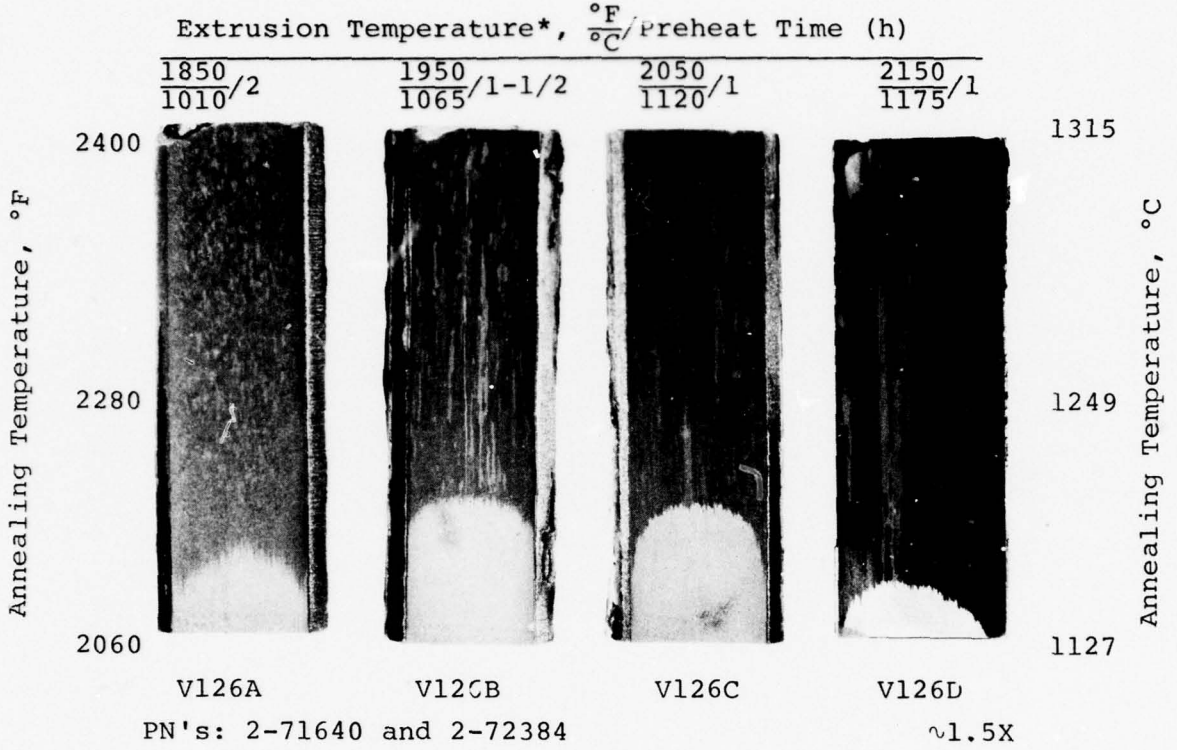


FIGURE 3: Macrographs of Alloy 38 (Ni-15At.%Cr-15.5At.%Al-2At.%W-1At.%Mo-0.5At.%Ta) extruded (*18:1 ratio) and gradient annealed bars.

Etchant: 45:45:10, $HCl:H_2O:H_2O_2$

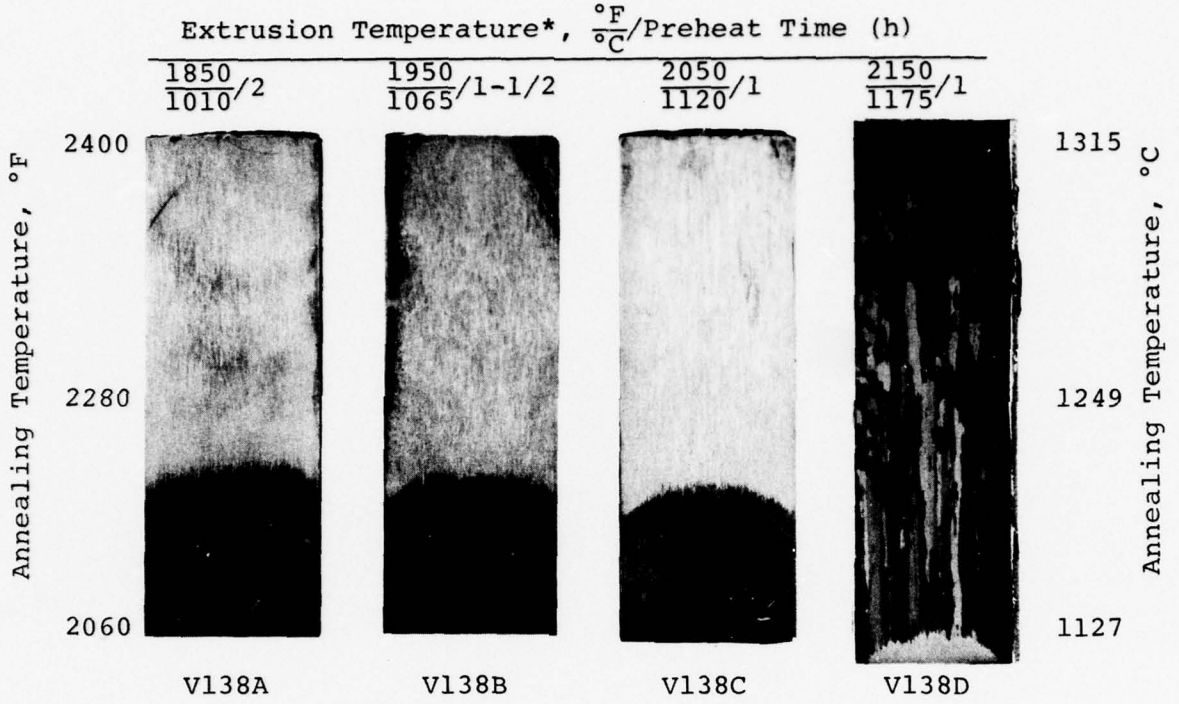


FIGURE 4: Macrographs of Alloy 42 (Ni-12.5At.%Cr-17At.%Al-2At.%W-1At.%Mo-0.5At.%Ta) extruded (*18:1 ratio) and gradient annealed bars.

Etchant: 45:45:10, $HCl:H_2O:H_2O_2$

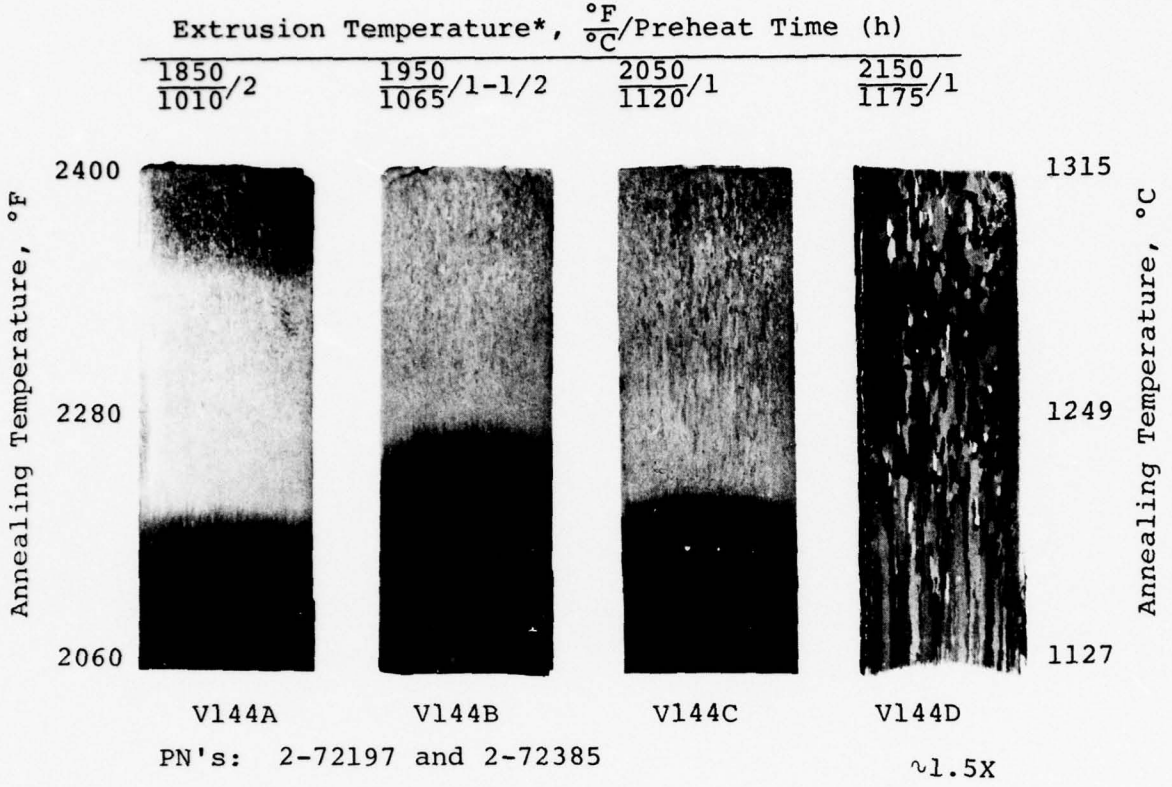


FIGURE 5: Macrographs of Alloy 44. Composition (wt.%): Ni-11.3Cr-7.3Al-6.4W-1.7Mo-3.2Ta-1.6Nb-1.1Y_2O_3. Extruded (18:1 ratio) and gradient annealed bars.

Etchant: 45:45:10, HCl:H_2O:H_2O_2

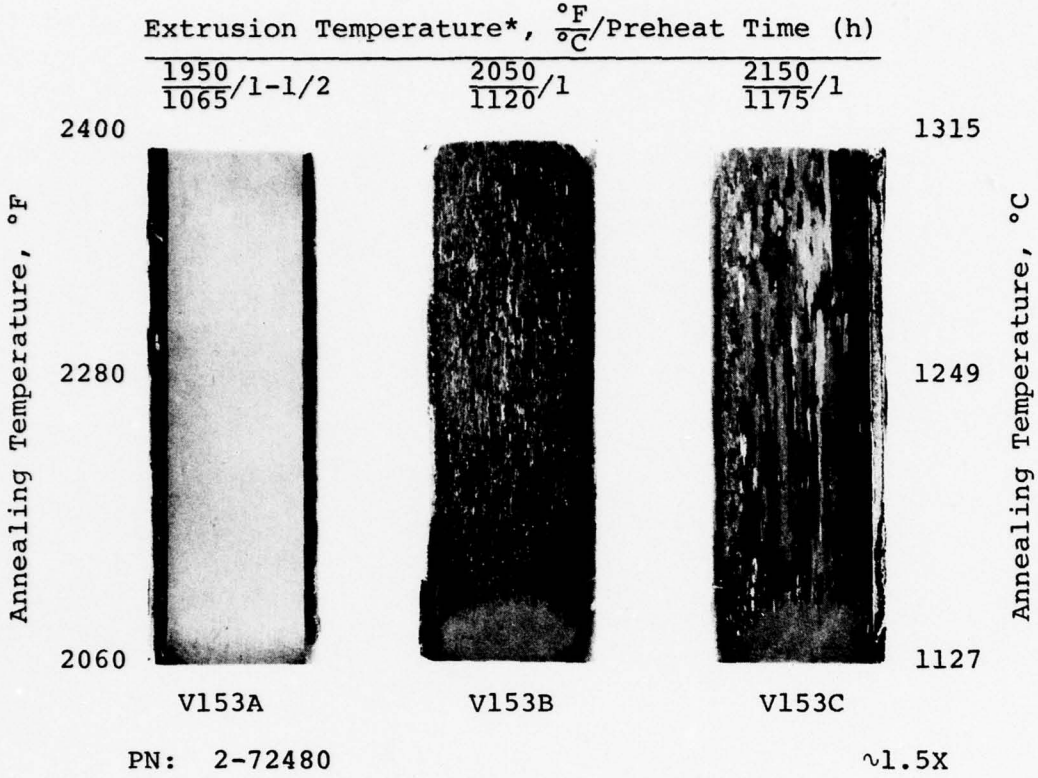
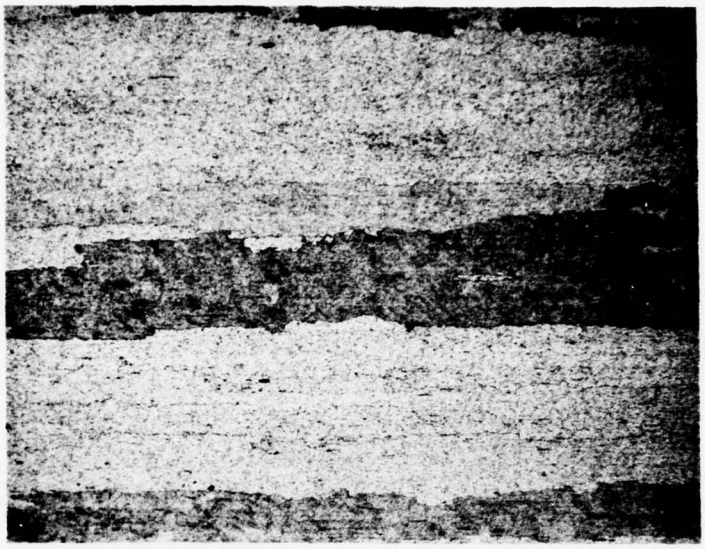


FIGURE 6: Macrographs of Alloy 47 (Ni-10At.%Cr-17.5At.%Al-2At.%W-2At.%Mo) extruded (*18:1 ratio) and gradient annealed bars.

Etchant: 45:45:10, $HCl:H_2O:H_2O_2$

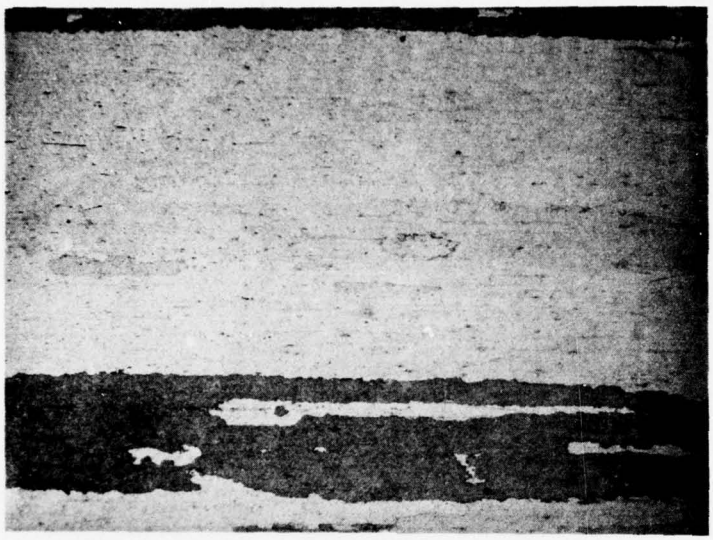


PN 1-72747 20X

FIGURE 7: Microstructure of Alloy 38. Bar V126D extruded 2150°F (1175°C)/18:1/3.6 ips (9.1 cmps) zone annealed 2360°F (1295°C)/2.8 iph (7.1 cmph).
Etchant: 45:45:10, $HCl:H_2O:H_2O_2$

PN 1-72748 20X

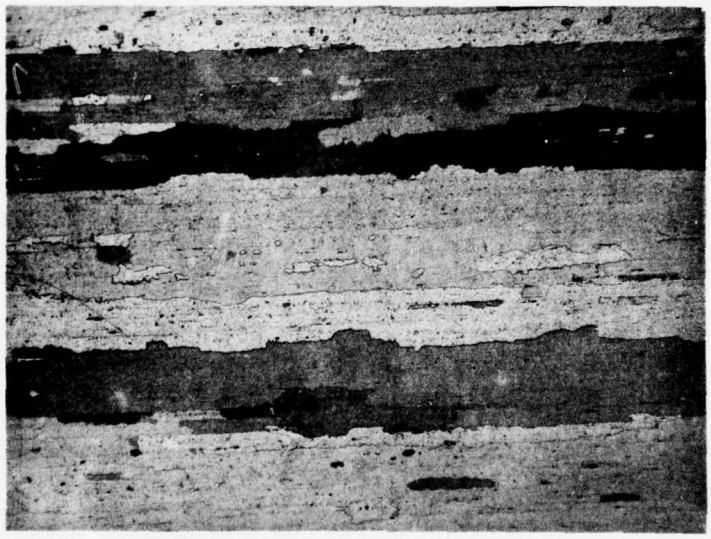
FIGURE 8: Microstructure of Alloy 42. Bar V138D extruded 2150°F (1175°C)/18:1/4.0 ips (10.2 cmps) zone annealed 2360°F (1295°C)/2.8 iph (7.1 cmph).
Etchant: 45:45:10, $HCl:H_2O:H_2O_2$



PN 1-72749 20X

FIGURE 9: Microstructure of Alloy 44. Bar V144B extruded 2150°F (1175°C)/18:1/4.0 ips (10.2 cmps) zone annealed 2360°F (1295°C)/2.8 iph (7.1 cmph).

Etchant: 45:45:10, $HCl:H_2O:H_2O_2$



PN 1-72751 20X

FIGURE 10: Microstructure of Alloy 47. Bar V153C extruded 2150°F (1175°C)/18:1/2.4 ips (6.1 cmps) zone annealed 2360°F (1295°C)/2.8 ips (7.1 cmps).

Etchant: 45:45:10, $HCl:H_2O:H_2O_2$

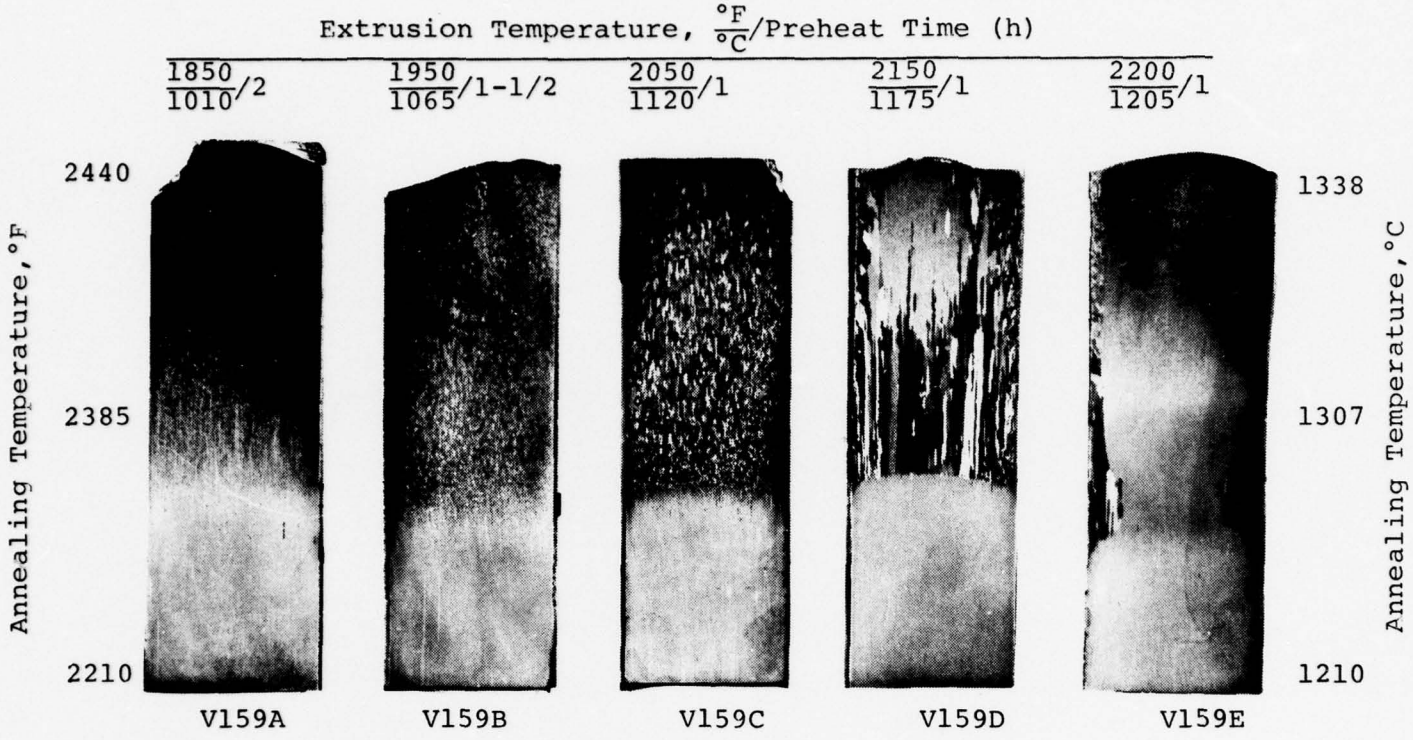


FIGURE 11: Macrographs of Alloy 49. Composition (wt.%): Ni-11.3Cr-7.3Al-6.4W-1.7Mo-3.2Ta-1.6Nb-0.15Zr-0.01B-1.1Y_2O_3. Extruded (18:1 ratio) and gradient annealed bars.

Etchant: 45:45:10, HCl:H_2O:H_2O_2

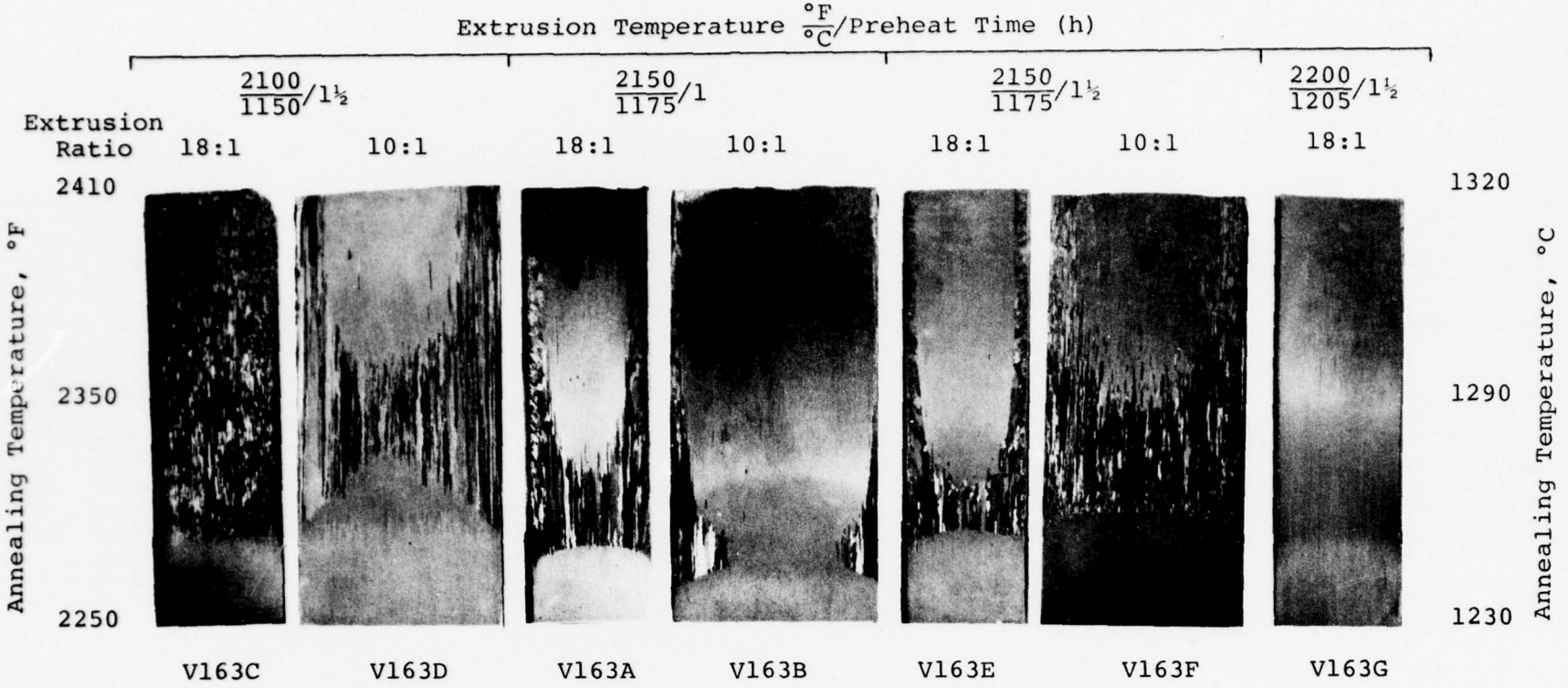


FIGURE 12: Macrographs of Alloy 49. Composition (wt.%): Ni-11.3Cr-7.3Al-6.4W-1.7Mo-3.2Ta-1.6Nb-0.15Zr-0.01B-1.1Y_2O_3

Extruded and gradient annealed bars.

Etchant: 45:45:10, HCl:H_2O:H_2O_2

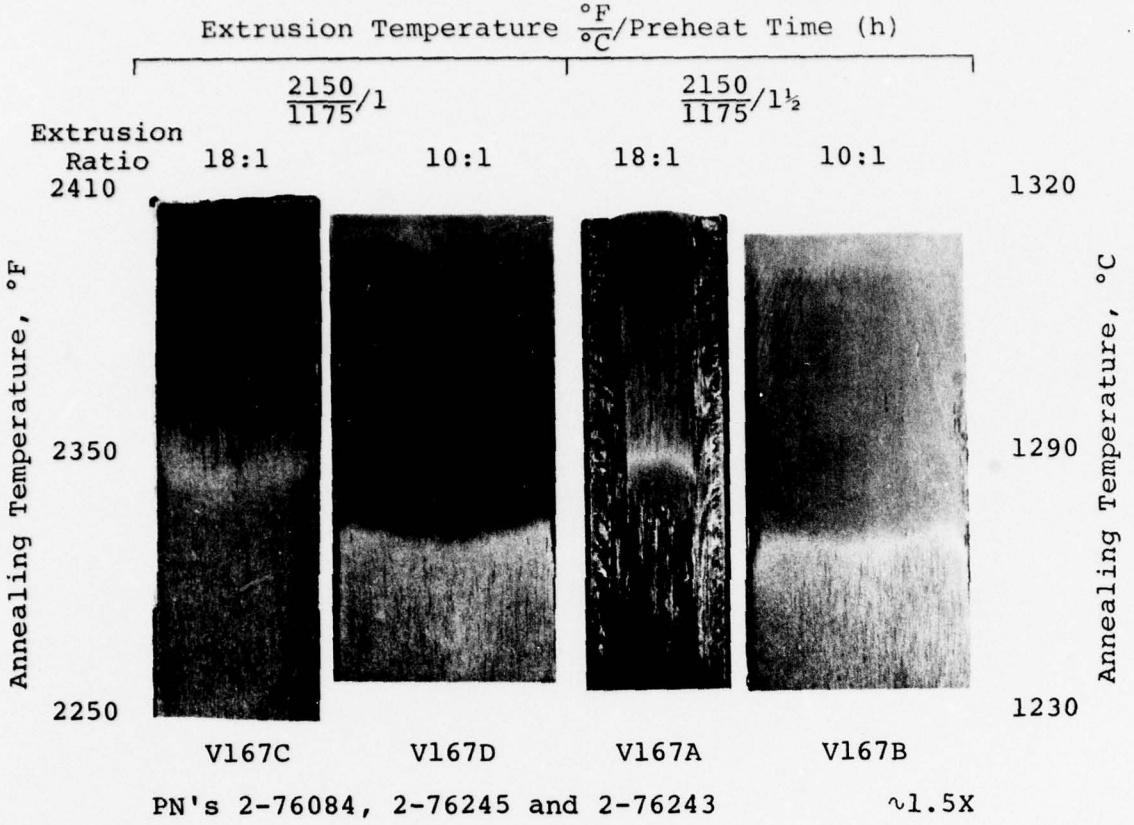


FIGURE 13: Macrographs of Alloy 50. Composition (wt.%): Ni-13.9Cr-7.4Al-6.5W-1.7Mo-1.6Ta-0.15Zr-0.01B-1.1Y_2O_3

Extruded and gradient annealed bars.

Etchant: 45:45:10, HCl:H_2O:H_2O_2

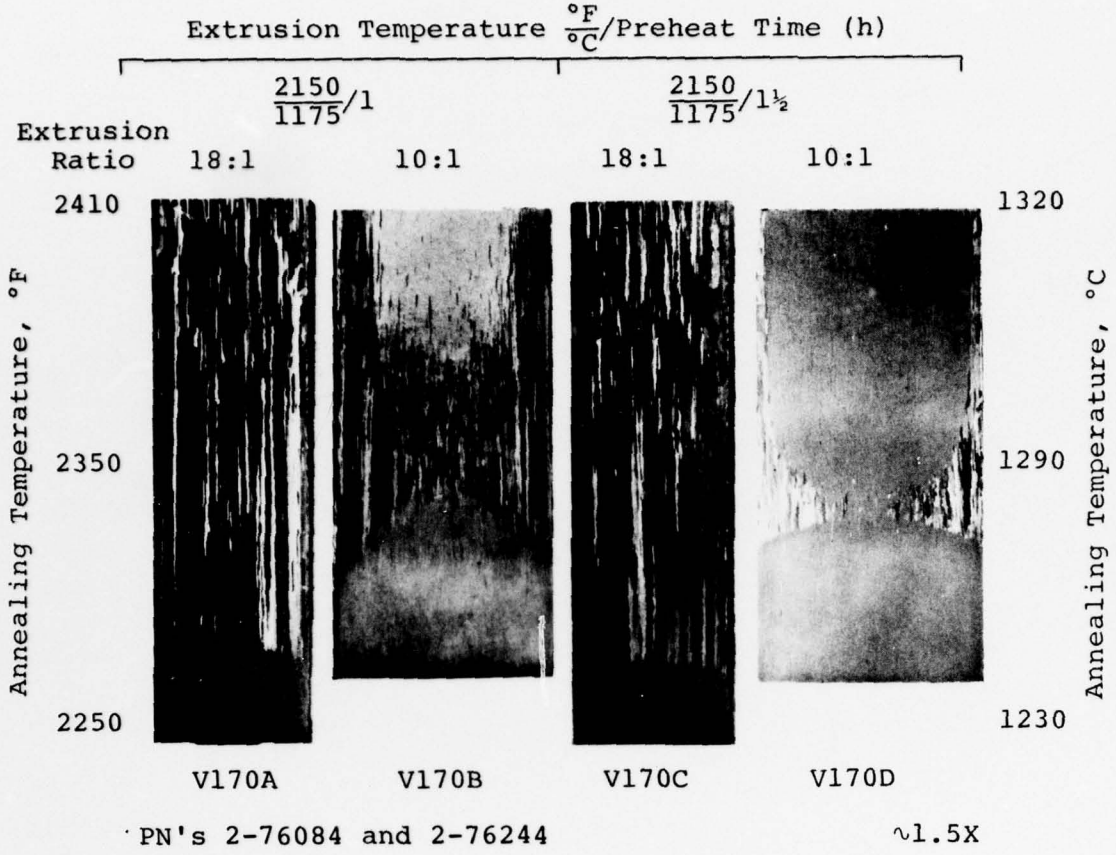


FIGURE 14: Macrographs of Alloy 51. Composition (wt.%): Ni-9.3Cr-8.5Al-6.6W-3.4Mo-0.15Zr-0.01B-1.1Y_2O_3

Extruded and gradient annealed bars.

Etchant: 45:45:10, HCl:H_2O:H_2O_2

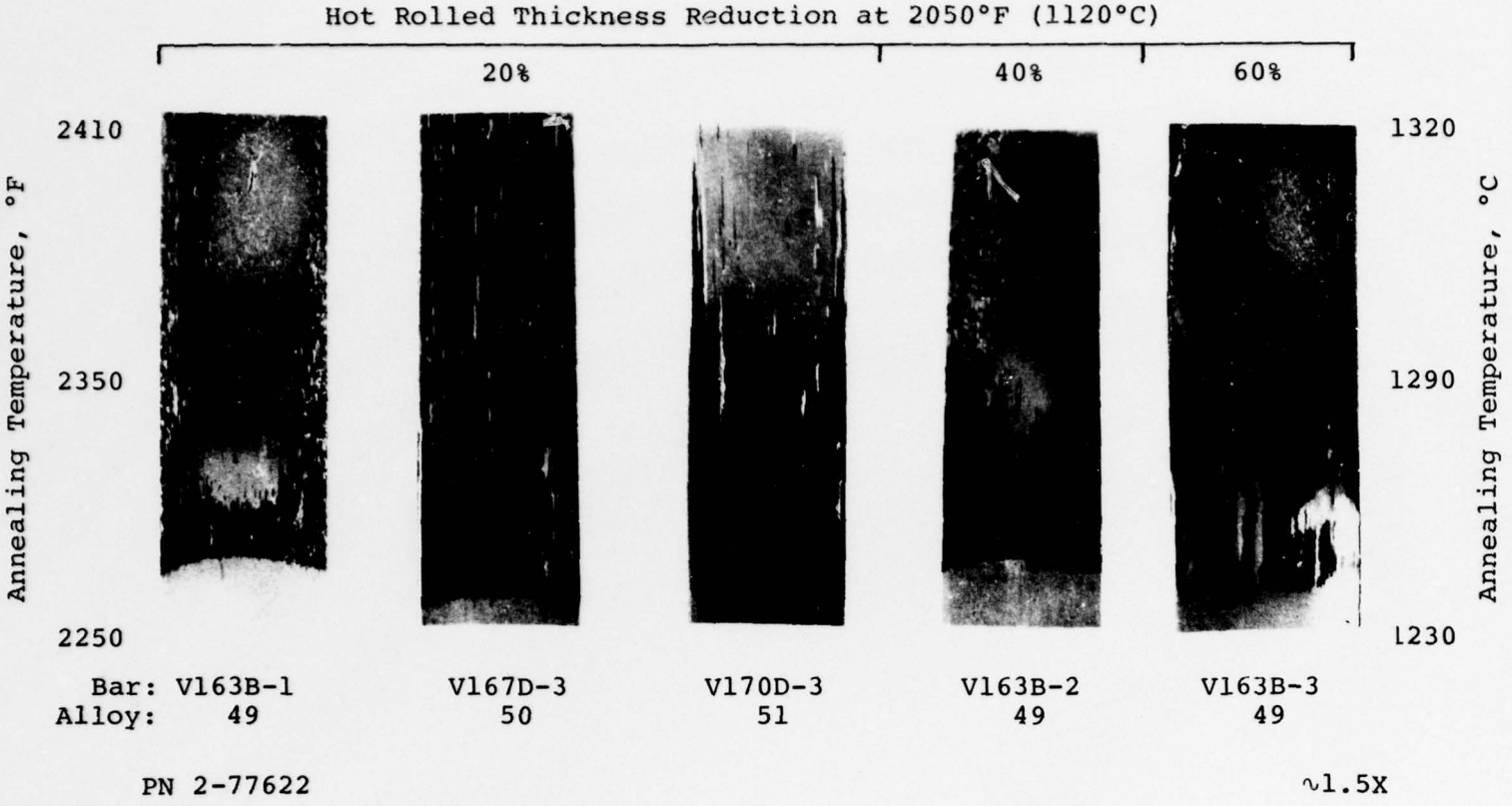


FIGURE 15: Macrographs of extruded, hot rolled and gradient annealed bars.
Composition (wt.%): Alloy 49: Ni-11.3Cr-7.3Al-6.4W-1.7Mo-3.2Ta-1.6Nb-0.15Zr-0.01B-1.1Y_2O_3

Alloy 50: Ni-13.9Cr-7.4Al-6.5W-1.7Mo-1.6Ta-0.15Zr-0.01B-1.1Y_2O_3

Alloy 51: Ni-9.3Cr-8.5Al-6.6W-3.4Mo-0.15Zr-0.01B-1.1Y_2O_3

Etchant: 45:45:10, $HCl:H_2O:H_2O_2$

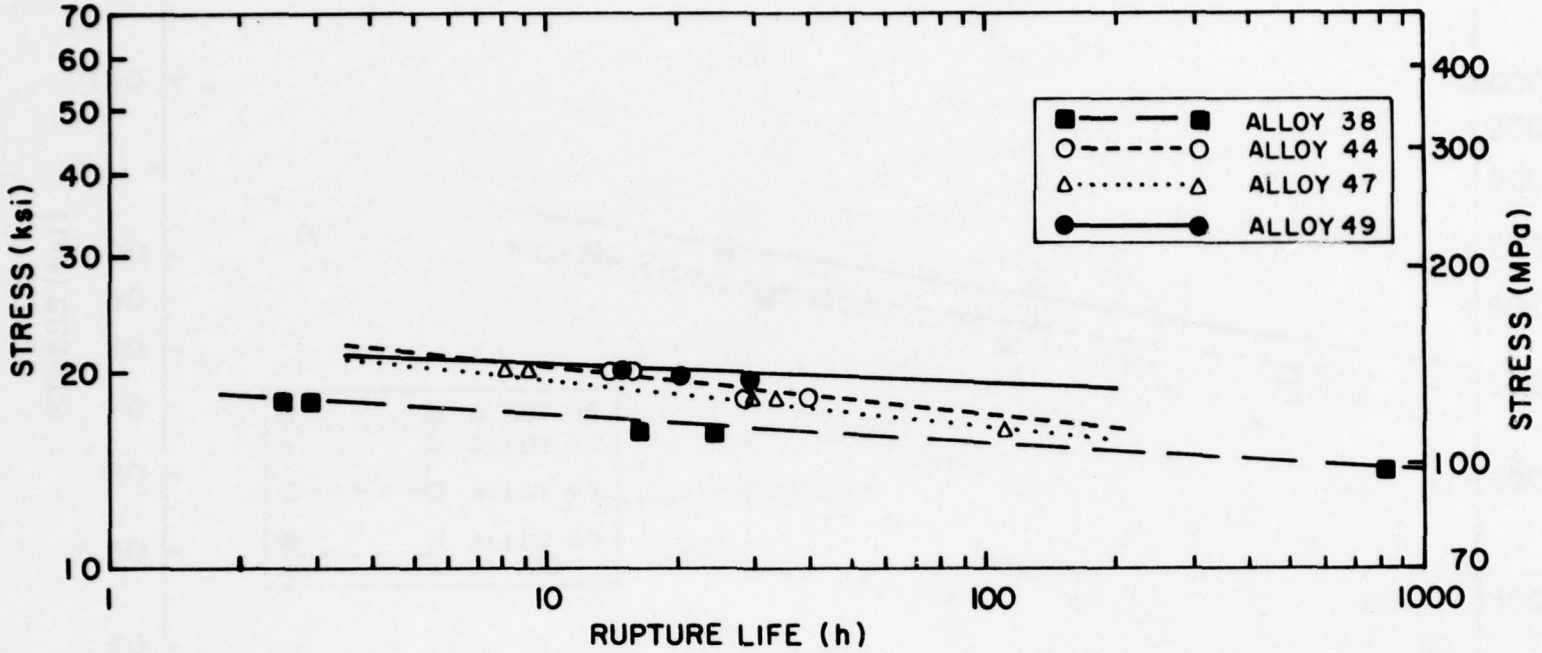


FIGURE 16 - 2000°F (1093°C) STRESS RUPTURE PROPERTIES OF ALLOYS 38, 44, 47 AND 49.

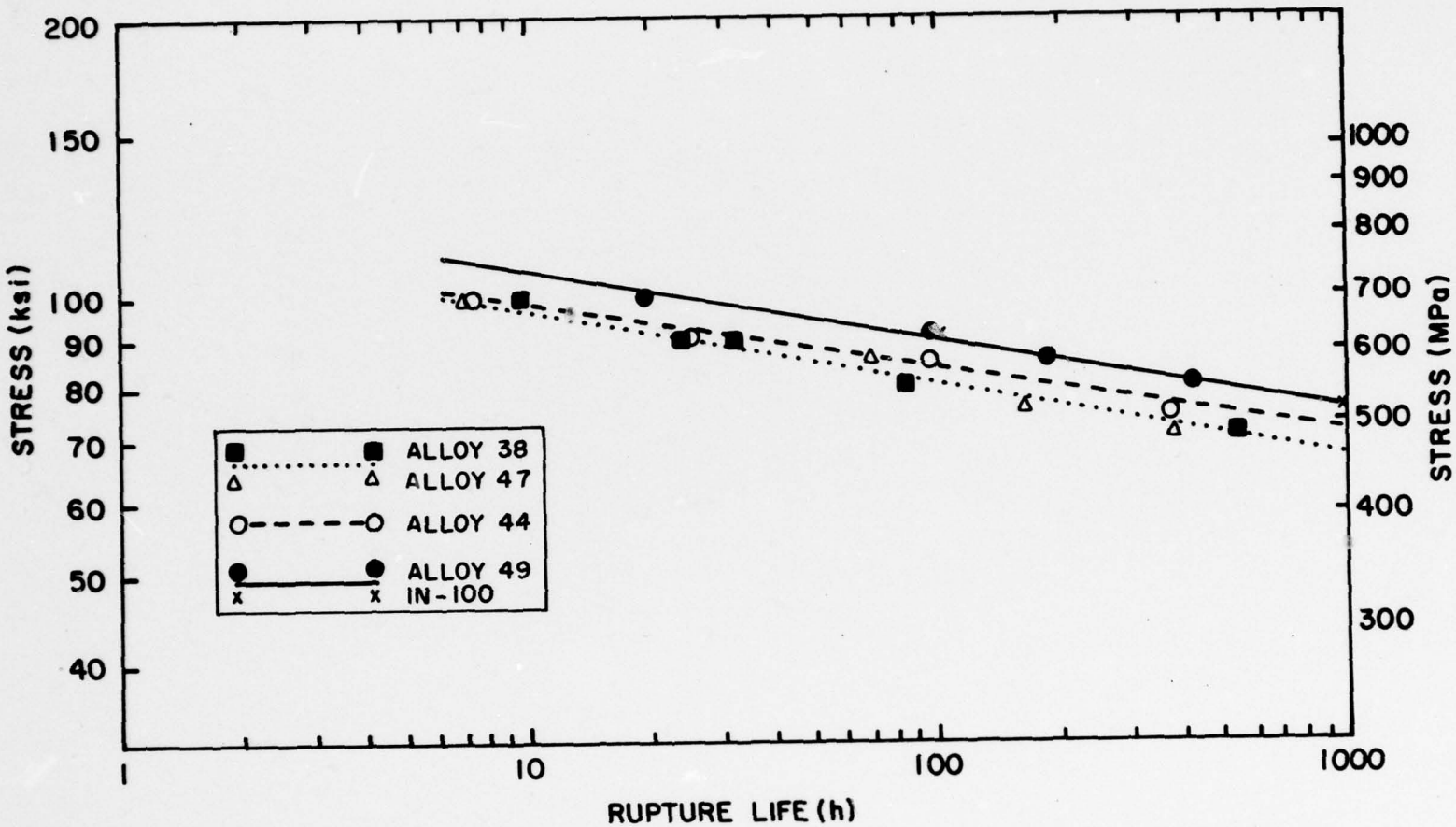


FIGURE 17 - 1400°F (760°C) STRESS RUPTURE PROPERTIES OF ALLOYS 38, 44, 47 AND 49.

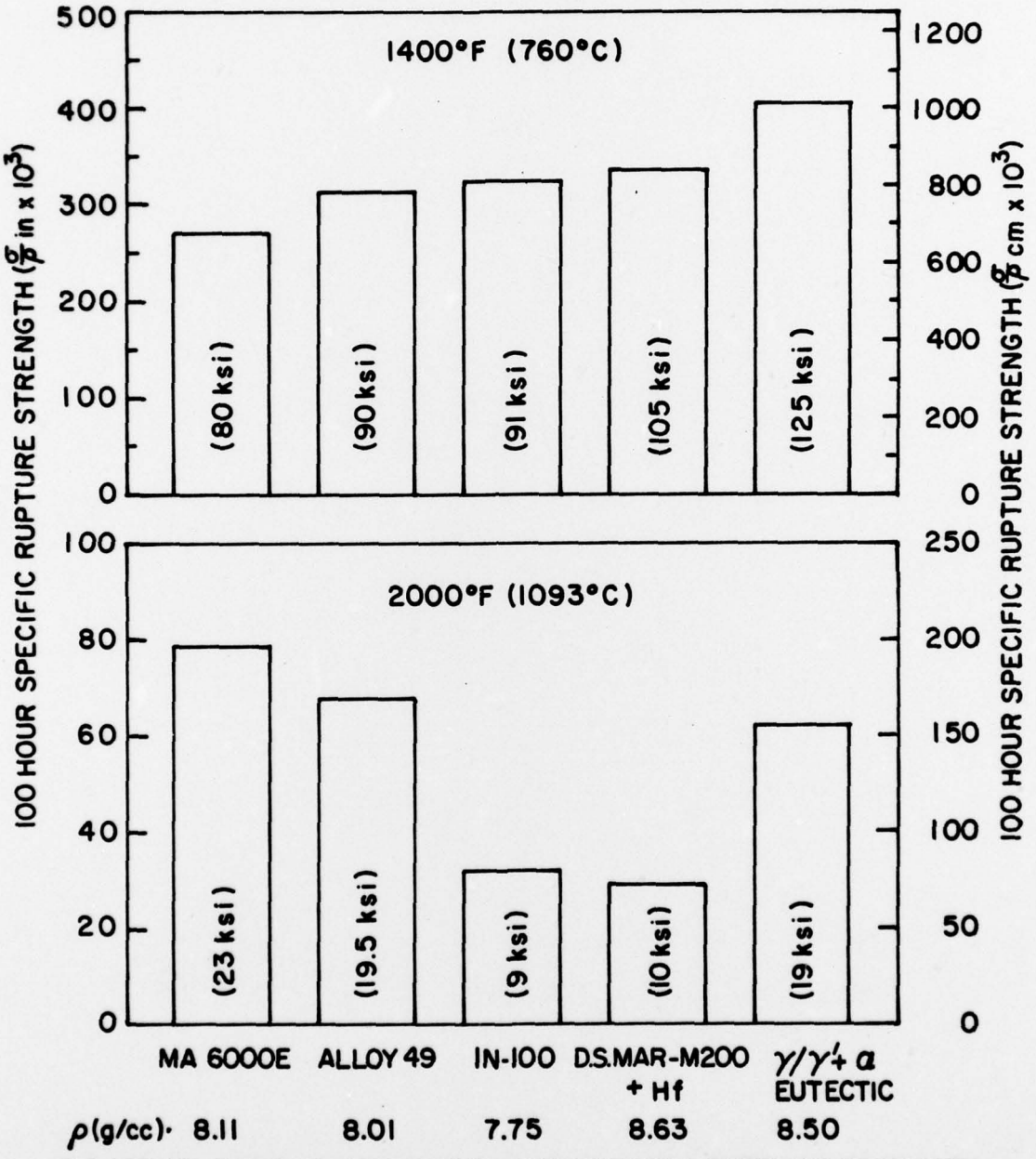
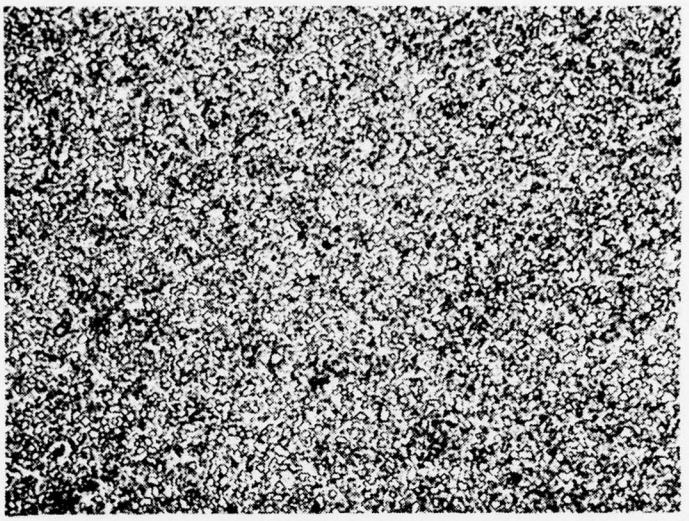
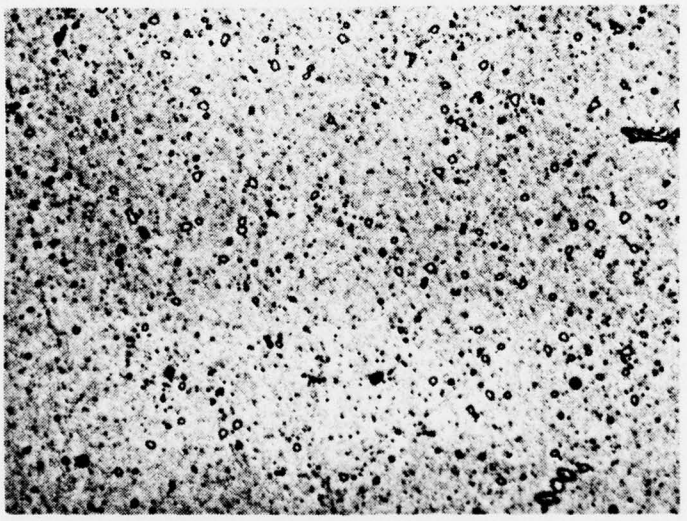


FIGURE 18 - COMPARISON OF 100 HOUR SPECIFIC RUPTURE STRENGTH CAPABILITY OF VARIOUS MATERIALS SYSTEMS.



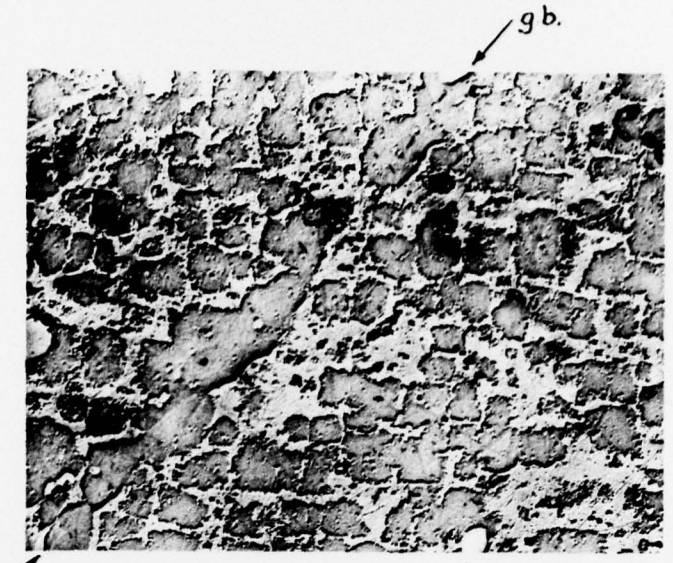
PN 1-74777 1000X

FIGURE 19: Microstructure of Alloy 44. Extruded bar V144D, zone annealed 2390°F(1310°C)/2.8 iph(7.1 cmph) and heat treated 2275°F(1245°C)/½ h/WQ.
Etchant: Glyceregia



PN 1-74623 1000X

FIGURE 20: Extruded and zone annealed as Figure 7, then heat treated 2300°F(1260°C)/½ h/WQ. Note reduction in γ' content.
Etchant: Glyceregia



EM 21065 4,900X

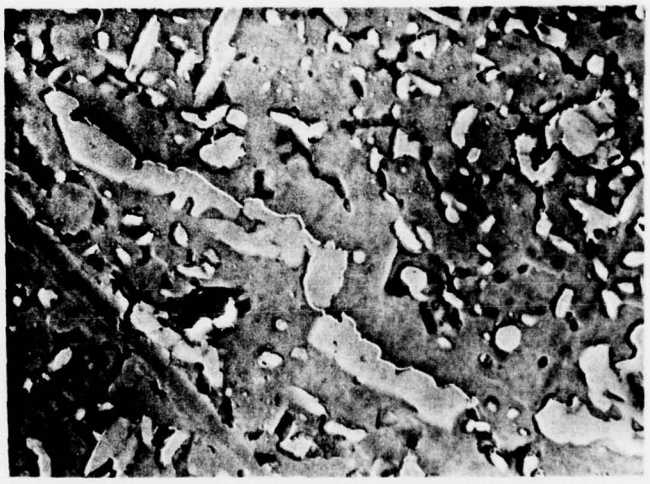
FIGURE 21: γ' morphology Alloy 44. Composition (wt.%): Ni-11.3Cr-7.3Al-6.4W-1.7Mo-3.2Ta-1.6Nb-1.1Y_2O_3. Extruded bar V144D, Z.A. 2360°F(1295°C)/2.8 iph (7.1 cmph) + 1/2 h/2360°F(1295°C)/AC.

EM 21067 4,900X

FIGURE 22: γ' morphology Alloy 49. Composition (wt.%): as Alloy 44 + 0.15Zr +0.01B. Extruded bar V159D, Z.A. 2340°F(1280°C)/2.8 iph (7.1 cmph) + 1/2 h/2340°F(1280°C)/AC.

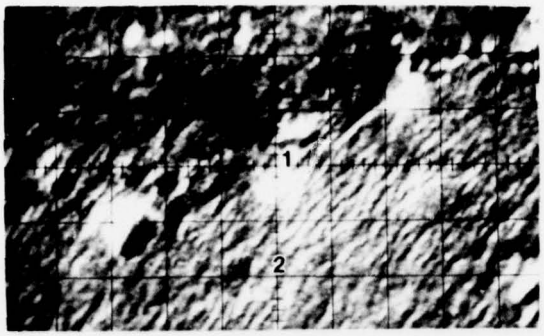
EM 21087 11,500X

FIGURE 23: Alloy 44, extruded bar V144D, zone annealed 2390°F(1310°C)/2.8 iph(7.1 cmph), heat treated 2 h/2360°F(1295°C)/FAC + 24 h/1660°F(905°C)/AC. Stress rupture: 1400°F(760°C)/85 ksi(586 MPa)/65 h.



EM 21094 11,500X

FIGURE 24: Alloy 49, extruded bar V159D, zone annealed 2370°F(1300°C)/2.8 iph(7.1 cmph), heat treated as above. Stress rupture: 1400°F(760°C)/85 ksi(586 MPa)/124 h.



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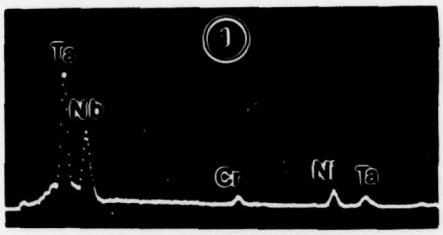

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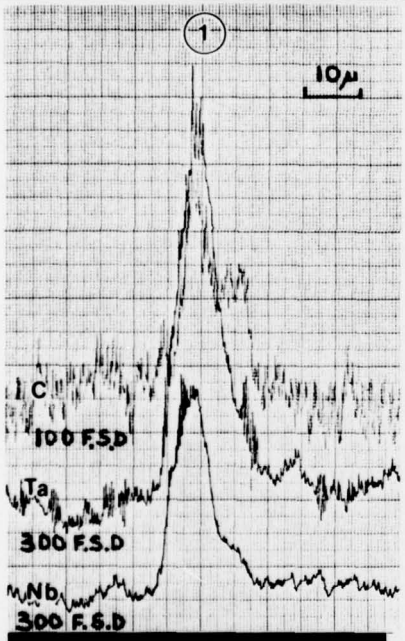

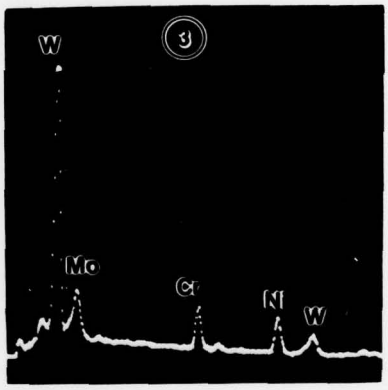

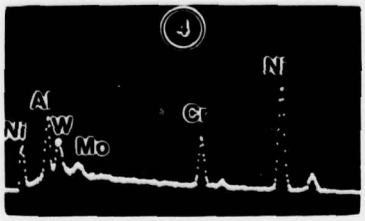

FIGURE 25: Electron microprobe analyses of alloy 49, zone annealed 2370°F(1300°C)/2.8 iph (7.1 cmph), heat treated A+B+C (see Table XIII) and rupture tested 1400°F(760°C)/85 ksi (586 MPa)/107-hour life. Micrographs show Ta/Nb-rich grain boundary carbides(1) and W/Mo-rich grain boundary and matrix carbides(3), compared to γ/γ' matrix(2)/(4).

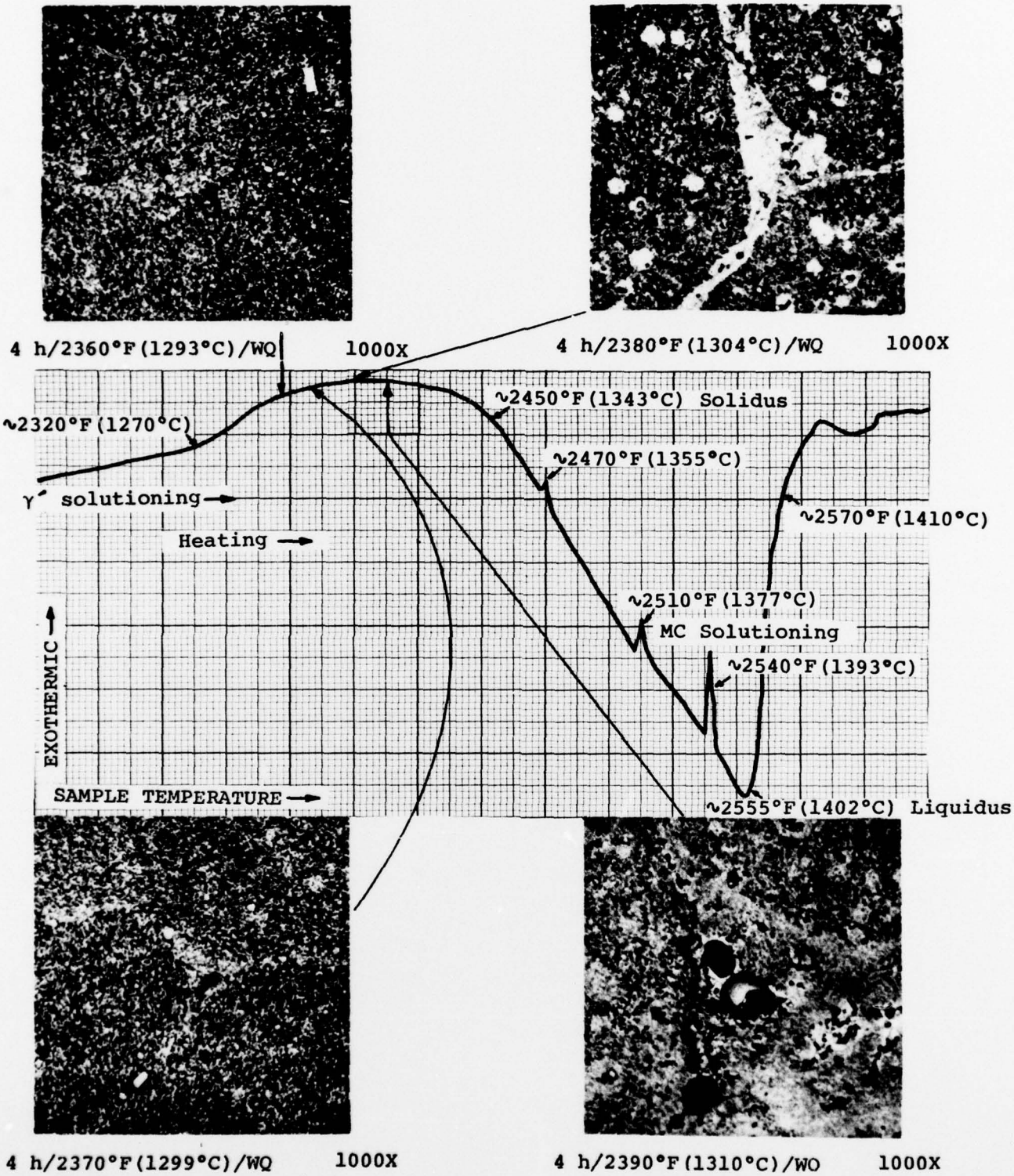

FIGURE 26: Differential thermal analysis trace of alloy 49.

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